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December 18, 2009

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Waste, Pesticides, and Toxics Division
U.S. Environmental Protection Agency
EPA Region 9
77 West Jackson Boulevard
Chicago, Illinois 60604-3590

Re: Quemetco, Inc., Indianapolis, Indiana (IND 000 199 653): Revised Workplan dated May 19, 2008: EPA Comments dated October 8, 2009

This letter responds to comments received from the US EPA in a letter dated October 8, 2009. In partial response to these comments, a sampling plan for XRF sampling was submitted to the U.S. EPA on December 11, 2009. (A revised more comprehensive plan for soil, sediment and surface water sampling will be completed based on the results of the XRF sampling. No sampling or testing will be conducted until agreement and approval is received from the US EPA on the proposed comprehensive Sampling and Analysis Plan (SAP). The revised QAPP is included as an attachment to this letter; the SAP will be submitted at a later date, following the XRF sampling, under separate cover.

A. ECOLOGICAL ISSUE COMMENTS: (2008)

The following comments are provided in review of pages 1-14 of the May 19, 2008 Workplan prepared by WSP to address outstanding DERA issues at the Quemetco Facility. In a letter dated July 24, 2008, Quemetco agreed to provide EPA with a figure showing all of the previous sampling locations, the concentrations of lead detected in those locations, and an indication of which locations exceed Region 5 recommended screening benchmarks, along with a topographic map.

Response: The referenced figure was submitted to the U.S EPA on April 14, 2009.

Upon review of these comments, and to assist EPA in its evaluation of Quemetco's progress in complying with the Consent Decree, Quemetco has agreed to discuss, with EPA any future proposed sampling locations.

Response: Agreed.

GENERAL COMMENTS:

1. Pursuant to the April 14, 1989, Consent Decree Civil Action No. IP87-684C, an Ecological Risk Assessment (ERA) was to be completed as part of the Resource Conservation and Recovery Act (RCRA) Facility Investigation Report (RFI) requirements. The RFI for the Site was *conditionally approved* on October 26, 1999. The conditions included Quemetco clearly defining the full nature and extent of the

contamination on Site as well as completing a human health and ecological risk assessment. To date, the full nature and extent of contamination at the Site has not been clearly defined and an ERA has not been approved.

✓ *Response: Agree. Additional soil, sediment and surface water sampling will be conducted to further delineate the nature and extent of contamination. WSP's proposed XRF sampling plan, dated December 11, 2009, has been submitted.*

2. Given the length of time that this project has been in progress, and to therefore avoid further iterations of sampling and analysis, EPA is requiring that the proposed soil, sediment and surface water sampling locations be expanded so as to more clearly define the nature and extent of contamination at the Site.

✓ *Response: A sampling plan for XRF sampling was submitted on December 11, 2009. A revised SAP for soil, sediment and surface water sampling will be completed based on the results of the XRF sampling.*

3. A figure showing all of the previous sampling locations, the concentrations of lead detected in those locations, and an indication of which locations exceed Region 5 recommended screening benchmarks, is requested to visually depict the extent of the lead contamination. In addition, a topographic map of the area showing surface water flow, including the location of Julia Creek as it parallels, enters, and exits the property, is requested. Quemetco has previously agreed to this request in a letter dated July 24, 2008. Once EPA has had an opportunity to review the impending information, along with the information contained in the SAP, sampling locations will be proposed by EPA for all future sampling efforts at the Site.

Inadequate?
✓ *Response: This has been completed and was submitted to the U.S. EPA on April 14, 2009. w/ Jennifer?*

4. Per the Ecological Risk Assessment Guidance for Superfund (USEPA, 1997), a field sampling plan (FSP) for an ecological investigation should include a description of the "sampling locations, timing, and frequency". "The FSP should be detailed enough that a sampling team unfamiliar with the site would be able to gather all the samples and/or required field data based on the guidelines presented in the document." Therefore, all of the soil, sediment, and surface water sampling locations will be proposed, and approved by EPA, prior to the sampling event. Sampling locations will be proposed based on the information provided in General Comment #3, as well as the information provided in this SAP.

✓ *Response: Agree.*

SPECIFIC COMMENTS:

5. Contents Page: The page numbers as listed do not match the text as provided. In addition, the pages are not numbered. Please revise.

✓ *Response: The page numbers on all documents have been reviewed and revised as necessary.*

6. Page 1, Section 1.0: Surface water samples will be collected from Pond #3 and Julia Creek for chemistry and toxicity testing using *P. promelas* and *C. dubia*, not only *P. promelas* as was indicated.

✓ *Response: Agree. Testing will be done on both species.*

7. Page 1, Section 1.0: Sediment and water sampling locations will be determined prior to the sampling event and will not "be determined in the field" as was indicated.

✓ *Response: Agree.*

8. Page 4, Section 2.1, Paragraph 2: Soil samples in the Northeast (NE) Area will be taken as proposed. In addition, soil sampling will continue further south, parallel to the boundary of the fence line, to the horizontal boundary of previous soil samples taken in that area. In addition, soil samples will also be taken to east of the proposed samples. The fact that the area to the east is characterized by heavily wooded areas does not preclude it from the investigation. Unless topography maps and surface run off patterns can be produced that suggest contamination from the NE Area is not affecting these wooded areas, nor Julia Creek, they will be investigated as part of the ERA. Since these lines of evidence have not been produced to date, additional soil samples will be taken from the fence line all the way to Julia Creek. Specific sampling locations will be selected based on the information provided in General Comment #3.

✓ *Response: Agree.*

9. Page 5, Section 2.2, Paragraph 3: The presence of the railroad in the area to the west of the Slag Pile does not preclude it from the investigation. Previous soil samples to the west of the Slag Pile had lead concentrations as high as 36,100 mg/kg. The lateral extent of the lead contamination to the west of the Slag Pile needs to be determined to rule out the off-site migration of lead. Additional soil samples to the west of the Slag Pile will be taken. Specific sampling locations will be selected based on the information provided in General Comment #3.

✓ *Response: Agree.*

10. Page 5, Section 2.3, Paragraph 1: Soil samples around the three Ponds will be taken as proposed. In addition, soil sampling will occur to the east of Ponds 1 and 2, where soil may have been impacted from contamination in the NE area. Previous sampling in the NE area suggests a south/southeasterly flow of contamination and previous samples collected to the east of Pond 1 had lead concentrations as high as 2,190 mg/kg. Therefore, the lateral extent of the lead contamination to the east of Ponds 1 and 2 needs to be determined. Additional soil samples to the east of Ponds 1 and 2 will be taken. Specific sampling locations will be selected based on the information provided in General Comment #3.

✓ *Response: Agree.*

11. Page 5, Section 2.3, Paragraph 1: One soil sample to east of Pond 3, as is proposed, will not fully characterize the area. Therefore, additional soil samples are required to the south of Pond 3 and the swale to more fully determine the extent of the contamination and to rule out the off-site migration of lead. Specific sampling locations will be selected based on the information provided in General Comment #3.

Response: Agree.

12. Page 5, Section 2.3, Paragraph 2: Soil samples along Julia Creek will be taken as proposed with the following additions. The sampling and analysis of soils along Julia Creek will be expanded to the northeast, off-site, where on-site contaminants from the NE area could be influencing Julia Creek. In addition, soil samples will be taken at points where the Creek goes off-site and/or is no longer in the path of influence from site contaminants to rule out off-site migration of contaminants. Specific sampling locations will be selected based on the information provided in General Comment #3.

Response: Agree.

13. Page 7, Table: The statement "All analyses will be performed using the following methods:" is not clear. The ICP-MS method 200.8 is intended for aqueous samples, while the SW-846 method 7471 is tailored for more solid matrices. This table should be corrected to specify which method will be used for which media.

Response: The revised table is presented as Table 1-1 in the QAPP.

14. Page 8, Section 3.0, Paragraph 3: In a December 12, 2007 letter, EPA concluded based on its review of the 2006 sampling data, that the DERA results from the toxicity tests indicated that Julia Creek water is highly toxic to *C. dubia* for the reproduction endpoint. Based on this conclusion, additional chronic toxicity tests using *C. dubia* and *P. promelas* will be conducted using Julia Creek water.

Response: Agree. Testing will be conducted on both species.

15. Page 8, Section 3.0, Paragraph 3: Surface water sampling locations will be determined prior to the sampling event and will not "be determined in the field by WSP representatives" as was indicated. See General Comment #4. In addition to the six samples proposed, EPA is also requiring additional surface water samples of Julia Creek where on-site contaminants from the NE area could be influencing the Creek. Specific sampling locations will be selected based on the information provided in General Comment #3.

Response: Agree.

16. Page 8, Section 3.0, Paragraph 4: The surface water samples collected from Pond 3 and Julia Creek will also be analyzed for water hardness.

Response: Agree.

17. Page 10, Section 4.0, Paragraph 2: Sediment sampling locations will be determined prior to the sampling event and will not "be determined in the field by WSP representatives" as was indicated. See General Comment #4.

Response: Agree.

18. Page 12, Section 5.0, Paragraph 1: The locations of the on-site and background soil samples to be used in the earthworm toxicity testing are not indicated in any of the attached Figures. Please include a Figure showing all of these proposed locations.

Response: Agree. The figure is included as Figure 2.

19. Page 13, Section 6.0, Paragraph 2: It is correct to assume that an individual Indiana Bat could obtain less than 10% of its diet from the site, based on the average foraging area of an Indiana bat. However, please include effects to the Indiana Bat based on it obtaining 100% of its diet from the site as well. This data would have been included had the Indiana Bat been included in the Screening Ecological Risk Assessment.

Response: This will be completed in the revised DERA.

B. QUALITY ASSURANCE DOCUMENTS (ECOLOGICAL) COMMENTS: (2008)

General:

There are many items left unaddressed by the submitted document revision. Internal referencing of appendices seems confusing or problematic. Laboratory and method selection(s) have not been fortified with sufficient rationale. The QAPP seems to have been assembled using older Agency guidance, while omitting key informational components that would properly document the basis for data collection. This QAPP is not ready for Agency approval.

Response: The QAPP has been completely re-written and conforms to the standards in EPA's May 1998 guidance document and includes all of the comments presented by EPA in this letter. The revised QAPP is included as an attachment.

Specific:

- 1.) Work Plan Section 2.0: Suitable and convincing rationale should be provided in an appropriate section of either the Work Plan or QAPP, supporting the particular chemicals of concern and test parameters identified in Table 1 which have been selected for this study. Why is data specifically needed for these chemicals and test parameters?

Response: Chemicals of concern were selected based on the results of the ecological risk assessment (see Cantox 2006 report). The chemicals listed are those which were not eliminated as chemicals of concern in the ERA. Other test parameters (e.g., hardness, total organic carbon) were selected to assist in the interpretation of toxicity test results.

- 2.) Work Plan Section 2.2: A better explanation is needed as to why samples need not be collected west of the railroad bordering the slag pile.

Response: This will be addressed in the revised SAP that will be submitted at a later date.

- 3.) Work Plan Section 2.3: It is apparent that many samples shall be collected for lead analyses. All of these are intended to be analyzed at a fixed laboratory. First, what are the relevant lead in soil concentration values (i.e. "action levels") upon which environmental decision-making shall be founded? **Also, the work plan writers should consider utilizing field XRF to make rapid turn-around field decisions to better**

demarcate the extent of lead contamination. Then samples could be sent to the laboratory for verification purposes.

Response: The action levels for each parameter are presented in Table 1-2 of the QAPP (page 11 of 14). Additionally, XRF sampling will be conducted onsite. WSP's XRF Sampling Plan, dated December 11, 2009 has been submitted to EPA and is included as Appendix B in the attached QAPP.

- 4.) Work Plan Section 2.3: The reference to "organic matter" in the last sentence of this section should be made more specific. Is the test parameter intended to mean "total organic carbon"?

Response: Yes, the test parameter is total organic carbon. The statement will be changed in the revised SAP to be submitted at a later date.

- 5.) Work Plan Section 2.4: The table embedded within this section states that "All analyses will be performed using the following methods." Yet this is merely an abbreviated version of Table 1 which follows later.

Response: This table will be omitted from the revised SAP that will be submitted at a later date.

- 6.) Work Plan Section 2.4: Many of the methods listed in the embedded table are water methods, when the heading section title refers to Soil Sample Collection. Other methods intended for soil samples that are indicated in Table 1 which follows are not indicated in this abbreviated table.

Response: This table will be omitted from the revised SAP that will be submitted at a later date.

- 7.) Work Plan Section 3.0: In the last par. of this section a reference to "interstitial pore water ammonia" is made in context of a test parameter. To my knowledge, SOPs have not been provided for this analysis, including sample preparation. Also, for purposes of this study, how is "interstitial pore water" defined?

Response: The SOP has been included in Appendix C of the QAPP (attached). The definition of interstitial pore water will be included in the revised SAP. However, for the purposes of this project, the ammonia will be analyzed in the water trapped in the unsaturated zone of several locations throughout the site.

- 8.) Work Plan Section 4.0: This section mentions phenol as an intended compound of concern for sediment analyses, but instead are other phenols needed for reporting purposes? Method 8270C can report a variety of phenolic compounds.

Response: Method 8270C will be used.

- 9.) Work Plan Section 4.1: In the individual bullets sometimes soil is referred to, and at other times 'sediment.' The subheading to this section regards "Sediment Sample Collection," so the individual bullets should be restated correctly and consistently.

Response: This section should only refer to sediment and will be corrected accordingly.

- 10.) Work Plan Section 4.1: In the 3rd par. from the end it states that the sediment samples will be "transported to the laboratory." It should be stated which laboratory shall receive these samples. (And 'ditto' for each of the sample groups.)

Response: The laboratories will be included in the revised SAP submitted at a later date.

- 11.) Work Plan Section 4.1: In the last par. of this section it is stated that a sketch will document each of the sample locations, which in this day & age sounds rather unsophisticated. Why not rely on a GIS/GPS technology system instead?

Response: Measurements will be taken in the field to properly identify all sample locations. The specific method to be used will be identified in the revised SAP to be submitted at a later date.

- 12.) Work Plan Section 5.0: The area designated as 'northeast area' could be distinguished more effectively from other surrounding areas on maps. It remains unclear as to where sample locations COMP 1 through COMP 7 mentioned in Table 1 are with respect to other locations shown on the map diagrams. If the metals to be analyzed in conjunction are those listed in Table 1, first row, and then it should be so stated in this narrative portion of the Work Plan.

Response: This information will be added to the revised SAP.

- 13.) Work Plan Section 6.0: Assessment of the Indiana Bat is not included in Table 1. Assumptions stated in this section must be assessed and confirmed by an eco-toxicity expert.

Response: Ruth Hull of Intrinsik Environmental Sciences has a MSc in ecotoxicology and over 18 years of experience in the field of ecotoxicology and ecological risk assessments. She prepared this section of the SAP.

- 14.) Table 1: Several SOPs are missing from the 'Method' column. __What is meant by the term, "as minimum" in the 1st column of the 9th row?

Response: Additional SOPs have been included and are now presented as Table 1-1 and are also included in Appendix C of the QAPP. The term "as minimum" has been removed from the table and additional metals to be analyzed have been included.

- 15.) Table 2: This Table is incomplete, as it only reflects metals analyses. There would be QA/QC procedures associated with the other test parameters as well. Will equipment rinse blanks of any kind also be used?

Response: This information is presented in more detail in Table 3-1 of the revised QAPP (Section 3, page 5 of 6). Equipment rinse blanks will not be used since all materials used to collect samples will either be sent to the laboratory or discarded after one-time use of collecting a sample.

- 16.) QAPP: Generally, this document might benefit from splitting out the chemistry tests versus the eco-bio-TOX type tests given that the QA/QC for each is relatively different. Just something for consideration.

Response: The chemical and ecological-biological-toxicology testing information and protocols have been separated in the revised QAPP.

- 17.) QAPP Title Page: More signature spaces are needed, including those for each of Quemetco's subcontracted laboratories. See the 1998 Region 5 RCRA QA Policy for further guidance.

Response: The signature spaces have been added to the revised QAPP along with additional EPA Region 5 RCRA personnel as requested in an email to WSP dated November 24, 2009.

- 18.) QAPP Appendices: The apparent trail of references to the various appendices, sometimes incorporated within appendices is very confusing to reviewers. Furthermore, it seems that several of these items were not submitted for review. The QAPP writers should go over these references again, sorting them out and ensuring that they're all present & accounted for. Otherwise, review of this QAPP cannot be conducted.

Response: The Appendices have been reviewed and revised accordingly.

- 19.) QAPP Section 1, Page 3 of 5: First, I couldn't find Table 1-2. Secondly, the term and values associated with 'EDQLs' have long since been superseded by 'Ecological Screening Levels,' or 'ESLs' a table for which may be found at the Region 5 RCRA corrective action website. It is essential that a table be prepared and submitted comparing the 'action' or decision levels,' (appropriately defined to meet particular specific project objectives for each chemical of concern, respectively), to analytical reporting limits for each proposed method. That way, methods which seem insufficiently sensitive can be screened out from the analytical program. Also, the rationale for each of the decision levels could be identified either in a column of the table or in footnotes to the table.

Response: An updated Table 1-2 is included in the attached revised QAPP (Section 1, page 11 of 14). It is recognized that EDQLs were replaced by ESLs. The ERA began before ESLs were put in place in 2003 (the revised PERA/SERA report was submitted in 2001). The rationale for the chosen decision levels has been described in detail in Section 1.4, page 10 of 14.

- 20.) QAPP Section 1.3.3, page 5 of 5: Where is Table 1-1 and how does this correspond to tables within the Work Plan? (Also, in regard to a Table needed for this QAPP, see preceding comment no. 19.)

Response: Table 1-1 is included in QAPP Section 1, page 2 of 14.

- 21.) QAPP Section 2.0: Here, an Appendix A is referenced that seems to match with a submitted hard copy of WSP's SOPs, (as opposed to what is stated here or in the Table of Contents).

Response: The appendices and SOPs included in the QAPP have been reviewed and revised. The correct appendices are indicated in the Table of Contents.

- 22.) QAPP Section 2.2, page 2 of 7: It should be stated here that this person shall perform data validation (if that is the case). See QAPP Section 8.2.

Response: This statement has been made and is now included in QAPP Section 2.3.1, page 2 of 8.

- 23.) QAPP Section 2.3, page 2 of 7: A table should be added clarifying which laboratory will be performing what specific analytical testing, respectively. Otherwise, it is all too confusing. The concept of the advertised Table 2-2 is well taken, but I did not receive a copy of this table to review. Following, then this QAPP section should be rewritten for *each* laboratory engaged in data collection activities, reflecting their own individual internal organizations & interfacing.

Response: A table clarifying the analytical testing that will be performed by each specific laboratory is included as Table 2-2 in QAPP Section 2, page 6 of 8. Section 2.4 of the QAPP has been re-written to include individual internal organizations for each laboratory.

- 24.) QAPP Section 2, page 6 of 7: Can Mr. Johnson take responsibility for validating ALL types of test data, including the eco/bio-TOX type tests? It would seem that different skill sets may be needed to handle the variety of data that will be generated for this study. Also, on this same page, will phenol(s) possibly be reported as 'TICs'? Of what use will TICs data be when the 8270C scan will be focused in on phenol(s) only? I wonder whether a 8270C-SIM scan should be done to enhance the reporting of phenols data? And, regarding the bullet stating 'data quality review,' does this mean 'data validation' or even suggest the presentation of a CLP-like Level 4 data package for each test parameter? (If not, please indicate in another QAPP section how thoroughly data quality shall be documented in the case of each test parameter?)

Response: Mr. Johnson is no longer employed with WSP. Ms. Terrie Baranek of ECT.Con, Inc. will assume the role of Quality Assurance Officer for the chemistry portion of the project and work closely with WSP. Ruth Hull of Intrinsik Environmental will be the Quality Assurance Officer for the eco-bio-tox portion of the investigation.

Phenols will be reported as TICs. The 8270C scan will be focused on phenols only. If analytical results indicate phenols are present in the sediments above the project action levels, than a more specific 8270C-SIM analysis will be conducted.

In response to your comment regarding data quality reviews, this has been further detailed in QAPP Section 9.2.

- 25.) QAPP section 3.0: What are the specific 'decision rules' for this project, defining how data shall be used to address specific project objectives?

Response: The decision rules for the project are described in detail in QAPP Section 1.4, page 9 of 14.

- 26.) QAPP Section 3.1, page 1 of 5: Table 3-1 wasn't provided. 'As-is,' however, this section as provided is relatively unhelpful for understanding the project, as is the case with sections on accuracy, completeness, and comparability. These are all just text book definitions that could be looked up. There should be a reemphasis here describing how individual data points will be utilized, as indicated in previous comments nos.19 and 25.

Response: Table 3-1 has been included in QAPP Section 3.7, page 5 of 6.

A detailed discussion of accuracy, completeness and comparability, including how data points will be utilized, is included in QAPP Section 3.

- 27.) QAPP Section 3.6: A table summarizing all QC sample types to be collected in conjunction with each test parameter (by analytical test & matrix), reflecting the sample network and design, should be submitted.

Response: Table 3-1 has been included in QAPP Section 3.7, page 5 of 6.

- 28.) QAPP Section 5.1: SOPs are needed for field data collection.

Response: All SOPs are included in Appendix C of the QAPP.

- 29.) QAPP Section 5.2: For this sort of information, specific sections corresponding to calibration procedures found within each respective laboratory SOP could be referenced. Note that submittal of Quality Program manuals shall not suffice for this purpose.

Response: The calibration procedures for each laboratory are included in QAPP Section 6.2, page 1 of 3, and in Appendix C.

- 30.) QAPP Section 6.0: A tabulation of all field testing to be conducted would nicely summarize the information called for in the first part of this section. Ditto for the laboratory methods for corresponding test parameters, by lab, respectively, & for each matrix type.

Response: Please see Tables 7-1 and 7-2 in QAPP Section 7, pages 1 of 2 and 2 of 2.

- 31.) QAPP Section 7.1: Section 5.0, as referenced, is effectively 'void.' Also, Table 3-1, referenced to section 3.0, was not provided.

Response: Section 5 has been incorporated into Section 6. Table 3-1 has been included in QAPP Section 3.7, page 5 of 6.

- 32.) QAPP Section 7.2: Here in the last bullet, graphite furnace is mentioned, but this method isn't mentioned in Table 1 of the Work Plan. Confusing.

② *Response: Graphite furnace has been removed.*

- 33.) QAPP Section 7.2, page 2 of 2: The reference to 'specific QC requirements,' is in & of itself insufficient and deficient. Per previous comment no.29, refer instead to specific SOP sections to address the relevant information. Will 'full CLP-type deliverable packages' be supplied in the case of all test parameters where data generation occurs at fixed laboratories (including each eco-bio-TOX test)? If not, then fuller explanation is needed? In the case of the 'phenol' test parameter, will full CLP-like results be reported for every SVOC on the method scan, or just for phenol (or just the relatively few phenols which can be reported via 8270C)?

Response: The reference to "specific QC requirements" has been removed. A detailed description of data reporting requirements is presented in QAPP Section 9.3.2, page 3 of 3.

- 34.) QAPP Section 8.2, page 2 of 4: For metals, furnace atomic absorption QC is stated, although this method isn't mentioned in Table 1 of the Work Plan. It is recommended that only data that will be generated for this study be included in this QAPP section (e.g. such as ICP-MS spectra for 6020, etc.). Also, phenol(s), ammonia, AVS-SEM and biota testing were omitted from this QAPP section.

Response: The reference to furnace atomic absorption has been removed. Data validation procedures are described in detail in QAPP Section 9.2, page 1 of 3. Table 3-1 has been revised to include all QAQC samples to be collected and analyzed (including those for phenols, ammonia and AVS-SEM). Table 3-1 is included in QAPP Section 3, page 5 of 6.

- 35.) QAPP Section 8.2, page 3 of 4: In reference to the phrase 'all data' stated on the first line on this page, I have the same concern as previously expressed, that not all test parameters might correspond to a CLP-like reporting format – in which case there should be further discussion.

Response: The statement "all data" has been removed. A detailed discussion of data validation and reporting is presented in QAPP Section 9.2 and 9.3, pages 1 and 3 of 3, respectively.

- 36.) QAPP Section 8.3, page 4 of 4: Insert the phrase, "Level IV" following the term, "CLP-like," found in the 4th line.

Response: The statement "Level IV" has been added. A detailed discussion of data reporting is presented in QAPP Section 9.3, page 3 of 3.

- 37.) QAPP Section 9.1: How do these procedures correspond to either non-CLP type testing or to data generated during bio-toxicity testing? Which tests are these definitions intended for?

Response: This section has been revised and is now presented in QAPP Section 12.

- 38.) QAPP Section 11, page 2 of 3: In the top line, the sentence fragment should be completed.

Response: This section has been revised and is now presented in QAPP Section 13.

- 39.) Comprehensive Quality Assurance Plan for Chemical Services (May 14, 2008): The problem with this document is that only test summaries are presented, rather than completely written, 'cookbook,' validated SOPs.

Response: Validated SOPs are included in QAPP Appendix C.

- 40.) "Appendix A" – WSP Environment & energy SOPs: How will collection and preparation of interstitial pore water be conducted leading to the analysis of ammonia? The SOP(s) should be submitted for review. Also, in the SOP for Surface Water and Sediment Sampling, collection of VOCs samples is mentioned. Just to be certain - VOCs aren't a part of this data collection activity, right?

Response: The SOP for preparation of interstitial pore water to be conducted is included in QAPP Appendix C.

Correct, VOCs are not part of this investigation.

- 41.) Inovatia Laboratories, Inc.: The problem with this document is that it is a quality assurance *program* plan, which doesn't include copies of complete, validated SOPs proposed for this study written in 'cookbook' format. And it is unspecific. A lengthy suite of organic compounds is listed within this document, when from reading of the Work Plan, only phenol is specifically needed.

Response: Not applicable. Inovatia Laboratories will not be used for this investigation. Details on each laboratory used for this investigation is included in Section 2.4, page 2 of 8.

C. SAMPLING PLAN FOR FURTHER CHARACTERIZATION:

The purpose of the following task described below is to lay out a phase -wise, organized approach for obtaining a complete, accurate picture of the site characterization, in order to evaluate future actions for corrective measures.

The following information describes a plan for conducting stratified random grid-spaced sampling of important land areas at the Quemetco facility. The grid spaced sampling would be used to facilitate further characterization of the site by employing X-ray fluorescence (XRF) on-site field sampling for lead coupled with the collection of an appropriate number of soil samples for confirmatory lab analysis.

The recommended grid spacing should not exceed 70 ft x 70 ft (4900 sq ft). The grids would be square shaped with the exception of areas where the presence of irregular site features (e.g., slag pile, ponds) could preclude a square shape.

Sampling Zones

(Please see the companion maps which delineate the Zones described below.)

Zone 1

Starting at the Northeast corner of the operating facility, construct a line from sample location HT-15 and running directly south to the Spill Run-Off Containment Trench. Then construct grids extending west from this line and continuing all the way to the Railroad berm. Then, on the west side of the operating facility, continue constructing grids moving south all the way to the Slag Pile and around the west side of the Slag Pile up to the Railroad Berm. Continue the grid around the Slag Pile south until two grid spacings beyond the south end of Pond #3.

Zone 2

Starting along the east side of the Slag Pile, construct a line extending from the north end of the Slag Pile to two grid spacings beyond the south end of Pond #3. Construct grids that continue east of this line until reaching Quemetco Drive (excluding the area actually covered by Pond #3).

Zone 3

On the east side of the facility, construct a line starting from sample location NE-SS-31 and extending directly east to the western bank of Julia Creek. Then construct grids extending south of this line and continuing all the way to the southern fence line of the operating facility.

Zone 4

Starting at a line along the southern-most grids in Zone 3, construct grids extending from Julia Creek on the east to Quemetco Drive on the west. Construct the grids to extend south to Pond #2 and around Pond #2 and continuing for two grid spacings south of Pond #2.

Sample Collection and Analysis

Samples collected for XRF in each of the four Zones described above and shown on the attached Map, should *alternate* from (1) surficial to (2) a 0 inch to 6 inch depth *plus* surficial (i.e. two XRF field measurements), based on ordering of field collection. Thus, for any Zone, in grid space # 1, only a surface soil lead reading would be taken. Then, in the next immediate grid spacing, both/(first) a surficial lead XRF reading would be recorded, followed by a measurement made on a field-homogenized sample taken from the 0 inch to 6 inch depth. Then, in the third sampled grid spacing, only a surficial XRF lead measurement would be made, and so on.

Five percent (5%) of sampled grids shall be further sampled for fixed-laboratory analysis of total lead content using an acceptable SW-846 guidance method (e.g. graphite furnace atomic absorption technique - GFAA). The reporting limit of the selected method should be less than targeted eco-risk and human health risk decision criteria. Each of the four gridded property "Zones" shall have five percent fixed-laboratory verification coverage. So, for example, if Zone 3 (northeast and east of facility operating fence line, to the west bank of Julia Creek) contains (circa) 40 grid spacings, then two verification samples shall be collected for fixed-laboratory analysis for total lead (e.g. using GFAA). These two samples should be collected in soil adjacent to the Julia Creek. For Zone 4, verification samples also should be collected along the west bank of the Julia Creek. For Zone 1, verification samples collected for fixed-laboratory GFAA analysis should be collected from grid spacings situated north and west of the operating fence line. For Zone 2, verification samples collected for fixed-laboratory GFAA analysis should be collected from grid spacings situated adjacent to Pond #3.


For optimal correlation, fixed-laboratory verification GFAA samples should be sub-sampled from the field-homogenized sample.

The XRF instrument should be employed in a manner consistent with SW-846, Guidance Method 6200 (Jan. 1998). Surficial XRF sampling should be performed as described in section 11.3 of Method 6200; intrusive 0 inch to 6 inch depth sampling for both XRF and GFAA purposes should be performed in accordance with section 11.4 of Method 6200. Exact sample locations should be recorded using a GPS device. The laboratory selected to perform GFAA analysis should be NELAC/NELAP - certified for lead analyses.

Response: A XRF Sampling Plan, dated December 11, 2009 has been submitted under separate cover.

Please don't hesitate to contact me if you have any questions or need additional information.

Sincerely yours,



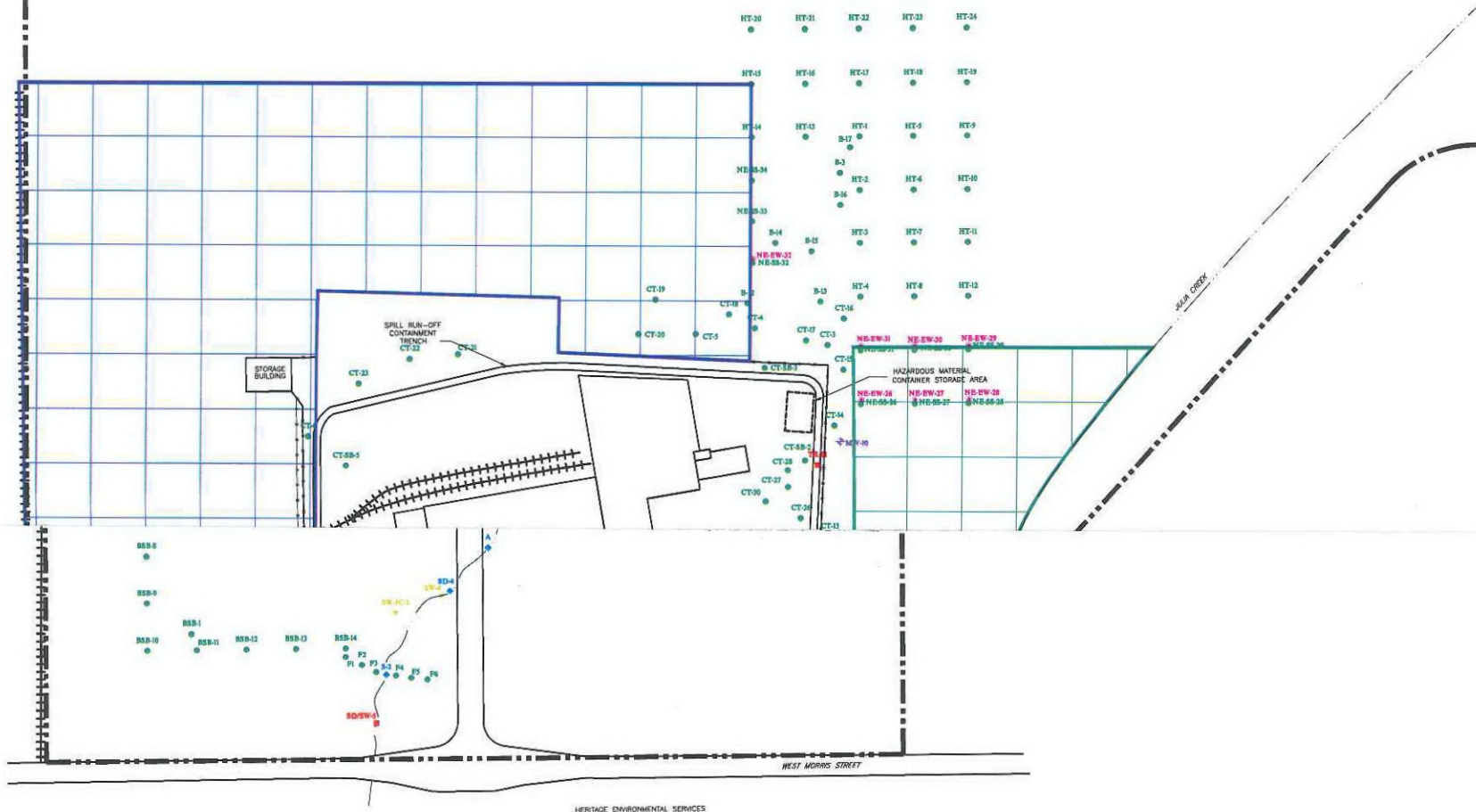
Richard E. Freudenberger
Vice President

REF:cv

Cc: Therese Cirone, RSR Corporation
Ruth Hull, Intrinsic Environmental Services, Inc.
Terrie Baranek, ECT.CON, Inc.



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SCALE IN FEET



WSP ENVIRONMENT & ENERGY
2025 GATEWAY PLACE SUITE 435
SAN JOSE, CALIFORNIA 95110
(408) 453-6100

SAMPLING GRID FOR XRF

900 QUEMETCO DRIVE
INDIANAPOLIS, INDIANA

PREPARED FOR
INDIANAPOLIS, INDIANA

DRAWN BY	JME
CHECKED	LB
APPROVED	
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SEAL
DATE

REVISIONS		
REV	DESCRIPTION	
1	Revised:	Chkd: Appr:
2	Revised:	Chkd: Appr:
3	Revised:	Chkd: Appr:

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FIGURE 2



Appendix A – WSP Environment and Energy's SOP

Standard Operating Procedure - 28

Screening of Soils for Metals using a Niton X-Ray Fluorescence Instrument

Materials:

Personal protective equipment (PPE)
Sample containers
Sample labels and indelible marker
Stainless steel trowels or spoons
Field log book
Niton XRF
Battery pack and charger
Shielded case
Calibration standards
Test platform
Ziploc bags
Test guard
Baby wipes
Sample tray

Note: Prior to obtaining a rental XRF unit, determine the requirements for radioactive material licensing and training from the State's Radiological Materials Department.

Calibration:

1. Every time the XRF is turned on or reset, the instrument performs a self calibration. In addition, several known standards are provided to check the calibration of the instrument. Calibrate the XRF at least daily, preferably each morning prior to conducting soil screenings, in the middle of the day, and at the end of day following soil screenings. Calibrate the XRF in accordance with the manufacturer's specifications using the low, medium, and high calibration standards.
2. Allow the instrument 15 minutes to warm up prior to field use.

In-situ Field Screening:

1. Select a measurement site. Always use the appropriate personal protective equipment based on the site conditions.
2. Clear any surface debris or vegetation from the measurement location, and ensure a flat surface to the soil. If necessary level the surface with a stainless-steel trowel or spoon.
3. Place the test guard on the ground.
4. Hold the Niton in one hand, and push the safety slide (that locks the shutter release) out from under the shutter release.
5. Place the Niton on the test guard so that the rectangular opening on the test guard is under the window of the Niton. Squeeze the shutter release and firmly press the instrument flat against the surface of the test guard (if you don't squeeze the shutter release, the plunger will not depress and the shutter will not fully open causing an inaccurate measurement). **Do not put your hand on**

the end plate of the unit while measuring. Never point the unit at yourself or anyone else when the shutter is open.

6. Watch for indications to decide when the test has reached the desired level of accuracy. A typical screening will last 30-60 seconds.
7. The instrument will provide a reading in ppm plus a measurement precision (+/-). Add the precision to the reading of concern to determine the metal concentration at that location.
8. Clean the bottom (soil contact side) of the Niton Test Guard with a baby wipe prior to the next screening location.

Note: In the unlikely event that the plunger gets stuck in the open position, simply push it closed. Then call the Niton Service Department at (401) 294-1234.

Ex-situ Field Screening:

1. Place the blade tip of trowel into the soil and push firmly to desired sampling depth.
2. Lift a portion of the soil out with the blade and place 50-100 grams of soil in a new plastic bag and close securely.
3. Label each bag with the sample location/ID.
4. Homogenize the sample in a plastic bag.
5. Shape the bag of soil to form a continuous uniform layer of at least one centimeter thickness and analyze the sample using the *in-situ* screening procedure (steps 3-8).

Note: Test results will tend to be 10-15% lower than results obtained using the *in situ* screening procedure.



Appendix B – Microbac Laboratories' SOP

STANDARD OPERATING PROCEDURE FOR METALS BY ICP-MS

Revision Author: Shon Ahrendt

This SOP is effective upon signed approval by the following:

Troy M. Boelt

8/25/2009

Inorganics Manager

Date

D.L.M.

QA/QC Manager

8/27/09

Date

DISCLAIMER: This SOP has been developed for use at the Microbac Laboratories, Merrillville, Indiana facility. It is intended for use by trained analysts. As written, this SOP may not be specifically applicable to the activities of other organizations.

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2.0 SCOPE AND APPLICATION

- 2.1 This is an Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) procedure for the determination of various metal elements. This procedure is applicable to the analysis of digestates from aqueous, non-aqueous liquid, drinking water, and solid matrix samples. The applicable analytes, detection limits and routine reporting limits (PQL) are listed at the Limits tab of the applicable test codes in LIMS.

3.0 SUMMARY

- 3.1 With the exception of some drinking water samples, samples must be digested using appropriate preparation methods. Digestion is not required when analyzing dissolved constituents provided the samples are filtered then preserved with acid. Digestion is not required when analyzing drinking water samples that have been determined to have a turbidity of <1 NTU.
- 3.2 Sample is nebulized into a spray chamber where a stream of argon carries the aerosol through a quartz torch and injects it into a radiofrequency plasma where the sample is decomposed and desolvated. The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that houses a quadrupole mass spectrometer. The ions are sorted according to their mass-to-charge ratio and measured with a detector.
- 3.3 This procedure allows for the determination of Mercury. A requirement for Mercury determination using ICP-MS is that the digestion procedure must use mixed nitric and hydrochloric acids through all steps of the digestion as Mercury will be lost if the sample is digested when hydrochloric acid is not present.
- 3.4 This procedure is based on the reference methods listed in section 17 of this document. This procedure contains no significant deviations from the reference methods. This procedure uses the following approaches, which are different than those found in the reference methods.
- 3.4.1 Section 9.2.3 of EPA Method 200.8 revision 5.4 requires the periodic analysis of a second source control standard (QCS). Section 9.3.4 of EPA Method 200.8 revision 5.4 requires the daily analysis of a calibration source control standard (IPC). This procedure combines the requirements of both these sections in a "more restrictive" approach by requiring the analysis of a second source control standard (ICV/CCV) on a daily basis.
- 3.4.2 Section 9.3.1 of EPA Method 200.8 revision 5.4 states, "When LRB values constitute 10% or more of the analyte level determined for a sample or is 2.2 times the analyte MDL whichever is greater, fresh aliquots of the samples must be prepared and analyzed again for the affected analytes after the source of contamination has been corrected and acceptable LRB values have been obtained". Most of the reporting limits (PQL) for this procedure are well above 2.2 times the MDL and meet the data quality objectives of the client. As such,

evaluation of the Method Blank down to 2.2 times the MDL is unnecessary as MBLK concentrations below the PQL are typically insignificant with respect to a detected concentration in a sample. Section 10 of this SOP requires that blanks are less than the PQL.

- 3.4.3 Based on the recommendation of the instrument manufacturer, Gold is added to the nitric acid as well as certain standards in an attempt to stabilize the Mercury concentration in samples and standards.
- 3.4.4 All digestates are diluted at least or minimally 1:5 for the initial analysis. This prepares the proper acid matrix allowing for a single digestate to be analyzed by ICP or ICP-MS.

4.0 DEFINITIONS

- 4.1 A list of definitions is in the Quality Assurance Plan. In addition to the terms defined in the QAP.

5.0 INTERFERENCES

- 5.1 Isobaric interferences occur when an isotope of one element is at the same nominal mass as an isotope of another element (e.g., Mo^{98} and Ru^{98}). Corrections for isobaric interferences may be made by measuring the intensity due to the interfering element at another isotope and using its natural abundance ratios to correct for its presence at the analytical mass of interest. With the exceptions of Molybdenum-98 (Ruthenium) and Selenium-82 (Krypton), most elements measured with this procedure have a minimum of one isotope free of isobaric elemental interference.
 - 5.1.1 Care should be taken that the isotope measured for correction purposes does not suffer from overlap with other isotopes that may be present in the sample.
- 5.2 Molecular interferences are caused by molecular species formed in the plasma with plasma or matrix ions (examples of common molecular interferences include ArCl , ClO , Nitrogen dimer, oxygen dimer, oxide species, double charged species, etc.). Predictions about the type of molecular interferences may be made using knowledge about the sample matrix. Molecular interferences can often be corrected for in the same manner as isobaric interferences, i.e., measuring the intensity present at another isotope and using isotope ratios to calculate the amount of the interfering species. For example, corrections for interferences of $\text{Ar}^{40}\text{Cl}^{35}$ on As at mass 75 may be made by measuring the intensity of ArCl present at mass 77 ($\text{Ar}^{40}\text{Cl}^{37}$) and converting to the apparent intensity of ArCl at mass 75 by using the isotopic ratio of Cl^{37} to Cl^{35} .
- 5.3 Abundance sensitivity is a property defining the degree to which the wings of a mass peak contribute to adjacent masses. The abundance sensitivity is affected by ion energy and quadrupole operating pressure. Wing overlap interferences may result when a small ion peak is being measured adjacent to a large one. The

potential for these interferences should be recognized and the spectrometer resolution adjusted to minimize them.

- 5.4 Physical interferences are associated with the physical processes which govern the transport of sample into the plasma, sample conversion processes in the plasma, and the transmission of ions through the plasma mass spectrometer interface. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension), or during excitation and ionization processes within the plasma itself. High levels of dissolved solids in the sample may contribute deposits of material on the extraction and/or skimmer cones reducing the effective diameter of the orifices and therefore ion transmission. These interferences are effectively removed through filtration of the sample digestate, and by matrix-matching the samples to the standards. Failure to meet the recovery criteria for the internal standards is indicative of these interferences and requires serial dilution analysis of the sample.
- 5.5 Carryover contamination is negated through the application of adequate rinse times between samples.
- 5.6 Additionally, solid phase chelation may be used to eliminate isobaric interferences from both element and molecular sources. There is an on-line that has been demonstrated for environmental waters such as sea water, drinking water and acid decomposed samples. Acid decomposed samples refer to samples decomposed by methods similar to methods SW-846 2052, 3051, 3050 or 3015. Samples with percent levels of iron and aluminum should be avoided. The method also provides a method for pre-concentration to enhance detection limits simultaneously with elimination of isobaric interferences. The method relies on chelating resins such as imminodiacetate or other appropriate resins and selectively concentrates the elements of interest while eliminating interfering elements from the sample matrix. By eliminating the elements that are direct isobaric interferences or those that form isobaric interfering molecular masses, the mass region is simplified and these interferences can not occur. The method has been proven effective for the certification of standard reference materials and validated using SRMs. The method has the potential to be used on-line or off-line as an effective sample preparation method specifically designed to address interference problems.

6.0 SAFETY

- 6.1 Consult the current revision of the Chemical Hygiene Plan. Requirements for the use of personal protective equipment (e.g. safety glasses, lab coats, gloves) as well as other area-specific safety requirements (e.g. gas cylinders) and MSDS sheets are addressed in the CHP.

7.0 EQUIPMENT AND SUPPLIES

The following is a list of materials needed to perform the steps of this procedure as written. See the reference method(s) for equipment and supply specifications.

- 7.1 All volumetric glassware/plasticware used shall be ASTM Class A. Class B glassware must be verified for accuracy on an annual basis and labeled with an appropriate correction.
- 7.2 PE Elan 9000 ICP-MS with a CETAC ASX-520 autosampler. The software steps in sections 11.0 and 12.0 are specific to the software used on the ELAN 9000 instrument.
- 7.3 Liquid Argon – 99.999% bulk liquid Argon, Krypton free
- 7.4 Polyscience recirculator model #3370
- 7.5 Adjustable and/or fixed volume micro-pipettes with tips to cover the range of 10 – 5000 µL
- 7.6 Digestion vessels, Environmental Express catalog #SC475 or equivalent; certified metals free. These vessels come with a Certificate of Accuracy from the vendor. This Certificate must be retained in order to show the volume verification and allowing them to be used without additional verification.
- 7.7 Filters, Environmental Express FilterMate, catalog #SC0401, or equivalent
- 7.8 Autosampler tubes: CPI catalog #4092-320 or equivalent
- 7.9 Clean 1-L plastic containers

8.0 REAGENTS AND STANDARDS

- 8.1 All reagents used must be analytical reagent (AR) grade or higher. All standards must be traceable to NIST, when available. Certificates of traceability must be obtained from the manufacturer. All reagents and standards must be documented in the appropriate preparation logbook. Refer to the requirements in the Labeling of Standards, Reagents, Digestates and Extracts SOP.

8.2 Reagents

All reagents are stored in the metals prep lab unless otherwise noted. Unless otherwise noted, prepared reagents are stored in appropriate plastic containers, retained in the metals prep lab and prepared on an as needed basis.

- 8.2.1 Lab pure water (DI water): Analyte free water is prepared as described in the Quality Assurance Plan. DI water may be obtained from any of the designated taps throughout the lab.
- 8.2.2 Hydrochloric acid, conc. (12N HCl): Metals grade, JT Baker Instra-analyzed reagent #9530-33 or equivalent.
- 8.2.3 Nitric acid, conc. (18N HNO₃): Metals grade, JT Baker Instra-analyzed reagent #9598-34 or equivalent.

- 8.2.4 Gold-stabilized Nitric acid, conc. (18N HNO₃) Add 0.75-ml of the 10,000 mg/L Stock Gold standard to each 2.5-L bottle of concentrated acid and label accordingly.

8.3 Standards

All standards are stored in the metals instrument lab unless otherwise noted.

8.3.1 Stock Standards

- 8.3.1.1 Stock Barium Tuning Standard, 1000-mg/L: Spex catalog # PLBA2-2T, or equivalent
- 8.3.1.2 Stock Beryllium Tuning Standard, 1000-mg/L: Elements catalog # Be1000-50
- 8.3.1.3 Stock Boron Calibration Standard, 1000-mg/L: CPI catalog # S4400-100074, or equivalent.
- 8.3.1.4 Stock Calcium Calibration Standard, 1000-mg/L: CPI catalog # S4400-100091, or equivalent
- 8.3.1.5 Stock Cerium Tuning Standard, 1000-mg/L: Elements Ce1000-50
- 8.3.1.6 Stock Cobalt Tuning Standard, Elements catalog # Co1000-50
- 8.3.1.7 Stock Germanium Internal Standard, 1000-mg/L: Elements catalog # Ge1000-50, or equivalent
- 8.3.1.8 Stock Gold Calibration Standard, 1000-mg/L: Elements catalog # Au1000-100, or equivalent
- 8.3.1.9 Stock Gold Standard, 10,000-mg/L: CPI catalog #S4400-10M212 or equivalent. The exclusive purpose of this standard is for it to be added to the concentrated Nitric acid.
- 8.3.1.10 Stock Indium Internal Standard, 1000-mg/L: Elements catalog # In1000-50, or equivalent
- 8.3.1.11 Stock Iron Calibration Standard, 1000-mg/L: CPI catalog # S4400-1000261, or equivalent
- 8.3.1.12 Stock Lead Tuning Standard, 1000-mg/L: CPI catalog # S4400-1000281
- 8.3.1.13 Stock Magnesium Calibration Standard, 1000-mg/L: CPI catalog # S4400-1000311, or equivalent. This may also be used as a tuning standard.
- 8.3.1.14 Stock Mercury Calibration Standard, 1000-mg/L: Spex catalog # PLHG4-24, or equivalent

- 8.3.1.15 Stock Mineral Calibration Standards, 1000-mg/L each: Spex catalog #'s CLFE2-24, CLCA2-24, PLK2-24, PLMG2-24, and PLNA2-24, or equivalent. These standards contain Fe, Ca, K, Mg and Na, respectively.
- 8.3.1.16 Stock Multi-element Calibration Standard, 100-mg/L each: CPI catalog # 4400-080111BD01 Microbac. This standard contains the following elements - Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Sn, Sr, Ti, Tl, V, Zn
- 8.3.1.17 Stock Palladium Calibration Standard, 1000-mg/L: Elements catalog # Pd1000-100, or equivalent
- 8.3.1.18 Stock Platinum Calibration Standard, 1000-mg/L: Elements catalog # Pt1000-50, or equivalent
- 8.3.1.19 Stock Potassium Calibration Standard, 1000-mg/L: Spex catalog # PLK2-24, or equivalent
- 8.3.1.20 Stock Rhodium Tuning Standard, 1000-mg/L: CPI catalog # S4400-1000442, or equivalent
- 8.3.1.21 Stock Scandium Internal Standard, 1000-mg/L: Elements catalog # Sc1000-50, or equivalent
- 8.3.1.22 Stock Sodium Calibration Standard, 1000-mg/L: Spex catalog # PLNA2-24, or equivalent
- 8.3.1.23 Stock Silver Calibration Standard, 1000-mg/L: Elements catalog # Ag1000-50 or equivalent.
- 8.3.1.24 Stock Terbium Internal Standard, 1000-mg/L: Elements catalog # Tb1000-50, or equivalent
- 8.3.1.25 Stock Uranium Tuning Standard, 1000-mg/L: Elements catalog # U1000-50, or equivalent

8.3.2 Intermediate Calibration Standards

- 8.3.2.1 Boron and Silver Intermediate Calibration Standard, 2.0-mg/L each: In a 50-ml digestion tube, dilute 0.1-ml of Stock Silver Calibration Standard and 0.1-ml of the Stock Boron Calibration Standard to the mark with 1-ml Gold-stabilized HNO₃ and DI water.
- 8.3.2.2 Gold, Palladium, and Platinum Intermediate Calibration Standard, 2.0-mg/L each: In a 50-ml digestion tube, add 0.1-ml of each of the following three standards: Stock Gold Calibration Standard, Stock Palladium Calibration Standard, Stock Platinum Calibration Standard. Dilute to the mark with 0.75-ml of concentrated HNO₃, 0.5-ml of concentrated HCl, and DI water.

- 8.3.2.3 Mercury Intermediate Calibration Standard #1, 5.0-mg/L: In a 50-ml digestion tube, dilute 0.25-ml of the Stock Mercury Calibration Standard to the mark with 0.5-ml of the Gold-stabilized HNO₃, 0.5-ml of the concentrated HCl, and DI water.
- 8.3.2.4 Mercury Intermediate Calibration Standard #2, 0.5-mg/L: In a 50-ml digestion tube, dilute 5.0-ml of the Mercury Intermediate Calibration Standard #1 to the mark with 0.5-ml of the Gold-stabilized HNO₃, 0.5-ml of the concentrated HCl, and DI water.
- 8.3.2.5 Mineral Intermediate Calibration Standard, 100 mg/L each: In a 50-ml digestion tube, dilute 5.0-ml of the Calcium Stock Calibration Standard, 5.0-ml of the Iron Stock Calibration Standard, 5.0-ml of the Magnesium Stock Calibration Standard, 5.0-ml of the Potassium Stock Calibration Standard, and 5.0-ml of the Sodium Stock Calibration Standards to the mark with 1-ml of the Gold-stabilized HNO₃ and DI water.
- 8.3.2.6 Multi-element Intermediate Calibration Standard, 2.0-mg/L each: In a 50-ml digestion tube, dilute 1.0-ml of the Stock Multi-element Calibration Standard to the mark with 2.5-ml of the Gold-stabilized HNO₃ and DI water.

8.3.3 Working Calibration Standards:

- 8.3.3.1 **Working Calibration Standards for Boron, Mercury, Silver, and Metals from the Multi-element Standard:** Prepare the following dilutions in 50-ml digestion tubes. Dilute the stated volumes of Spike Standard, 0.75-ml of Gold-stabilized HNO₃, and 0.5-ml concentrated HCl to 50-ml with DI water. The standards are prepared as needed and have been shown stable for up to 2-weeks.

Cal Std	Volume and Type of Spike Standard	Final Hg Conc. (µg/L)	Final Conc., Other Metals (µg/L)
CAL BLK	• None added	0	0
CAL STD 0.2	• 0.5-ml CAL STD 2	0.04	0.2
CAL STD 1	• 0.025-ml Multi-element Int. Cal. Std • 0.025-ml Boron and Silver Int. Std • 0.020-ml Mercury Int. Cal. Std #2	0.2	1.0
CAL STD 2	• 0.50-ml Multi-element Int. Cal. Std • 0.5-ml Boron and Silver Int. Std • 0.20-ml Mercury Int. Cal. Std #2	2	20
CAL STD 3	• 2.5-ml Multi-element Int. Cal. Std • 2.5-ml Boron and Silver Int. Std • 0.5-ml Mercury Int. Cal. Std #2	5	100
CAL STD 4	• 2.5-ml Multi-element Int. Cal. Std	0	500
CAL STD 5	• 5.0-ml Mineral Int. Cal. Std	0	10,000

- 8.3.3.2 **Working Calibration Standards for Gold, Palladium, and Platinum.** Prepare the following dilutions in 50-ml digestion tubes. Dilute the stated volumes of Gold, Palladium, and Platinum Intermediate Calibration Standard, 0.75-ml of concentrated HNO₃, and 0.5-ml concentrated HCl to 50-ml with DI water. The standards are prepared as needed and have been shown stable for up to 2-weeks.

Cal Std	Volume of Standard (ml)	Final Conc. of Au, Pd, Pt (µg/L)
CAL BLK	0	0
CAL STD 1	0.025	1.0
CAL STD 2	0.50	20
CAL STD 3	2.5	100

- 8.3.4 **Stock Verification Standards:** All Stock Verification Standards must come from a different source than the equivalent Stock Calibration Standards.

- 8.3.4.1 Stock Boron Verification Standard, Elements catalog # B1000-50, or equivalent
- 8.3.4.2 Stock Gold Verification Standard, 1000-mg/L: CPI catalog # S4400-1000212, or equivalent
- 8.3.4.3 Stock Mercury Verification Standard, 1000-mg/L: CPI catalog #S4400-1000331 or equivalent
- 8.3.4.4 Stock Mineral Verification Standard, 200-mg/L each: Spex catalog #CL-ICS-3 or equivalent. This standard contains Fe, Ca, K, Mg and Na.
- 8.3.4.5 Stock Multi-element Verification Standard, 100-mg/L each: Perkin Elmer catalog # N9301721 or equivalent. This standard contains the following elements - Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Sn, Sr, Ti, Tl, V, Zn.
- 8.3.4.6 Stock Palladium Verification Standard, 1000-mg/L: CPI catalog # P/N S4400-1000381, or equivalent
- 8.3.4.7 Stock Platinum Verification Standard, 1000-mg/L: CPI# P/N S4400-1000402, or equivalent

8.3.5 Intermediate Verification Standards

- 8.3.5.1 Gold, Palladium, and Platinum Intermediate Verification Standard, 1.0-mg/L each: In a 50-ml digestion tube, add 0.05-ml of each of the following three standards: Stock Palladium Verification Standard, Stock Platinum Verification Standard, and Stock Gold Verification Standard. Dilute to the mark with 0.75-ml of concentrated HNO₃, 0.5-ml of concentrated HCl, and DI water.

- 8.3.5.2 Mercury Intermediate Verification Standard #1, 5.0 mg/L: In a 50-ml digestion tube, dilute 0.25-ml of the Stock Mercury Verification Standard to the mark with 0.5-ml of the Gold-stabilized concentrated HNO_3 , 0.5-ml of the concentrated HCl, and DI water.
- 8.3.5.3 Mercury Intermediate Verification Standard #2, 0.5 mg/L: In a 50-ml digestion tube, dilute 2.5-ml of the Mercury Intermediate Verification Standard #1 to the mark with 0.5-ml of the gold-stabilized concentrated HNO_3 , 0.5-ml of the concentrated HCl, and DI water.
- 8.3.5.4 Multi-element Intermediate Verification Standard, 1.0 mg/L: In a 50-ml digestion tube, dilute 0.5-ml of the Stock Multi-element Verification Standard and 0.05-ml of the Stock Boron Verification Standard to the mark with 2.5-ml of the Gold-stabilized HNO_3 and DI water.

8.3.6 Working Verification Standards

- 8.3.6.1 ICV for Boron, Mercury, Silver, and Metals from the Multi-element Standard: In a 50-ml digestion tube, dilute 1.25-ml of the Multi-element Intermediate Verification Standard, 0.25-ml of the Mercury Intermediate Verification Standard #2, and 0.5-ml of the Stock Mineral Verification Standard to the mark with 0.75-ml of the Gold-stabilized HNO_3 , 0.5-ml of the concentrated HCl, and DI water. Final concentrations are 25- $\mu\text{g/L}$ each except Hg which is at 1.25- $\mu\text{g/L}$ and Fe, Ca, K, Mg and Na which are at 2025- $\mu\text{g/L}$ each. Prepare as needed.
- 8.3.6.2 CCV for Boron, Mercury, Silver, and Metals from the Multi-element Standard: In a 50-ml digestion tube, dilute 2.5-ml of the Multi-element Intermediate Verification Standard, 0.5-ml of the Mercury Intermediate Verification Standard #2, and 1.0-ml of the Stock Mineral Verification Standard to the mark with 0.75-ml of the Gold-stabilized HNO_3 , 0.5-ml of the concentrated HCl, and DI water. Final concentrations are 50- $\mu\text{g/L}$ each except Hg which is at 2.5- $\mu\text{g/L}$ and Fe, Ca, K, Mg and Na which are at 4050- $\mu\text{g/L}$ each. Prepare as needed.
- 8.3.6.3 ICV for Gold, Palladium, and Platinum. In a 50-ml digestion tube, dilute 1.25-ml of the Gold, Palladium, and Platinum Intermediate Verification Standard to the mark with 0.75-ml of the concentrated HNO_3 , 0.5-ml of the concentrated HCl, and DI water. Final concentrations are 25- $\mu\text{g/L}$ for each metal.
- 8.3.6.4 CCV for Gold, Palladium, and Platinum: In a 50-ml digestion tube, dilute 2.5-ml of the Gold, Palladium, and Platinum Intermediate Verification Standard to the mark with 0.75-ml of the concentrated HNO_3 , 0.5-ml of the concentrated HCl, and DI water. Final concentrations are 50- $\mu\text{g/L}$ for each metal.

8.3.7 Interference Standards

- 8.3.7.1 Stock Interference Check Standard, ICSA: Environmental Express catalog #ICP-MS-ICSA or equivalent. This standard contains Al, Na, Ca, P, Fe, K, Mg and S at 500 mg/L each, C at 1000 mg/L, Cl at 3600 mg/L, and Mo and Ti at 10 mg/L each.

- 8.3.7.2 Working ICSA Standard for Boron, Mercury, Silver, and Metals from the Multi-element Standard: In a 50-ml digestion tube, dilute 5.0-ml of the stock ICSA standard to the mark with 0.5-ml of the Gold-stabilized concentrated HNO_3 , and DI water. This produces a standard containing Al, Na, Ca, P, Fe, K, Mg and S at 50 mg/L each, C at 100 mg/L, Cl at 360 mg/L, and Mo and Ti at 1.0 mg/L each.
- 8.3.7.3 Working ICSAB Standard for Boron, Mercury, Silver, and Metals from the Multi-element Standard: In a 50-ml digestion tube, dilute the following solutions to the mark with DI water: 5.0-ml of the stock ICSA; 2.5-ml of Multi-element Intermediate Verification Standard; 0.5-ml of Mercury Intermediate Verification Standard #2; and 0.75-ml of the Gold-stabilized concentrated HNO_3 . This produces a standard containing the same analytes and concentrations found in the working ICSA standard as well as all other analytes in method at working CCV standard concentrations.
- 8.3.7.4 Working ICSA Standard for Gold, Palladium, and Platinum: In a 50-ml digestion tube, dilute the following solutions to the mark with DI water: 5.0-ml of the stock ICSA standard; 0.5-ml of the concentrated HNO_3 ; 0.11-ml of the 10000 mg/L Gold standard. This produces a standard containing Al, Na, Ca, P, Fe, K, Mg and S at 50-mg/L each, C at 100-mg/L, Cl at 350-mg/L, and Mo and Ti at 1.0-mg/L each.
- 8.3.7.5 Working ICSAB Standard for Gold, Palladium, and Platinum: In a 50-ml digestion tube, dilute the following solutions to the mark with DI water: 5.0-ml of the Stock ICSA, 2.5-ml of Gold, Palladium, and Platinum Intermediate Verification Standard; 0.75-ml of concentrated HNO_3 . This produces a standard containing the same analytes and concentrations found in the working ICSA standard as well as Au, Pd, and Pt at 0.05-mg/L each.
- 8.3.8 Rinse Solution for Boron, Mercury, Silver, and Metals from the Multi-element Standard: Dilute 30-ml of the Gold-stabilized concentrated HNO_3 , 20-ml concentrated HCl , and 0.1-ml of the 10,000-mg/L Gold standard to 2-L with DI water.
- 8.3.9 Rinse Solution for Gold, Palladium, and Platinum: Dilute 30-ml of the concentrated HNO_3 , 20-ml concentrated HCl , and 0.1-ml of the 10,000-mg/L Gold standard to 2-L with DI water.
- 8.3.10 Working Internal Standard Solution: In a 1-L plastic container, dilute 0.06-ml of the Stock Indium Internal Standard, 0.06-ml of the Stock Terbium Internal Standard, 0.3-ml of the Stock Scandium Internal Standard, and 0.5-ml of the Stock Germanium Internal Standard to approximately 1-L with 15-ml of concentrated HNO_3 , 10-ml concentrated HCl , and DI water.
- 8.3.11 Tuning Solution: Perkin Elmer catalog #PE8125040 contains Ba, Be, Ce, Co, In, Mg, Pb, Rh, and U at 10 ug/L each.

- 8.3.12 ALTERNATE Tuning Solution: An alternative to using the purchased Perkin Elmer standard is to purchase individual stock standards at 1000 mg/L each of Ba, Be, Ce, Co, In, Mg, Pb, Rh, and U then perform the following dilutions in order to prepare a working tune solution.
- 8.3.12.1 ALTERNATE Intermediate Tuning Solution, 10 mg/L each: In a 50-ml digestion tube, dilute 0.5-ml of the stock Ba, Be, Ce, Co, In, Mg, Pb, Rh, and U standards to 50-ml with 1-ml of the gold-stabilized concentrated HNO_3 and DI water.
- 8.3.12.2 ALTERNATE Working Tuning Solution, 10- $\mu\text{g/L}$ each: In a 1-L plastic container, dilute 1-ml of the ALTERNATE Intermediate Tuning Solution and 10-ml of Gold-stabilized concentrated HNO_3 to the mark with DI water.

9.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

- 9.1 The client or other trained personnel collect samples. Samples received at the laboratory are considered representative unless otherwise noted.
- 9.2 Water samples should be collected in a plastic container. Chemical preservation consists of HNO_3 to pH <2 for water samples only. Samples are stored on the shelves in the metals preparation lab. Prior to sample preparation, samples must be held for a minimum of 16-hours after preservation. Solid samples should be retained at 0.1-6°C until prepared and are stored in the walk-in cooler located in the sample receipt area. Digestates are stored in capped digestion tubes and kept in the prep lab.
- 9.3 Analysis must be performed within the maximum allowable hold time of 6-months from collection with the exception of Mercury, which has a hold time of 28-days.

10.0 QUALITY CONTROL

- 10.1 An *Initial Demonstration of Capability* study must be performed prior to the initial analysis for each analyst and whenever substantial change has occurred in the procedure or instrument. Refer to the Capability and Detection Limit Studies SOP for details.
- 10.2 An *Instrument Detection Limit* study must be performed for each new procedure, every three months thereafter, and whenever a change in instrument occurs. Refer to the Capability and Detection Limit Studies SOP for details. IDL's are calculated using data from analyses on three non-consecutive days.
- 10.3 A *Method Detection Limit* study must be performed for each new procedure, annually thereafter, and whenever a change in instrument occurs. Refer to the Capability and Detection Limit Studies SOP for details.
- 10.4 *Pre-calibration Checks* must be performed daily prior to calibration. This encompasses an Instrument Tune (Mass Calibration Check and a Resolution Check) and a Daily Performance Check.

- 10.4.1 A *Mass Calibration Check* is performed using the Tune Solution. Acceptance criteria are a mass cal value within ± 0.1 amu from the "true" unit mass. If reanalysis fails to meet the acceptance criteria, stop analysis, perform appropriate instrument maintenance then repeat.
- 10.4.2 A *Resolution Check* is performed using the Tune Solution. Acceptance criteria are 0.65amu at 10% peak height, which is equivalent to 0.75amu at 5% peak height. If reanalysis fails to meet the acceptance criteria, stop analysis, perform appropriate instrument maintenance then repeat.
- 10.4.3 The *Daily Performance Check* is performed by analyzing the Tune Solution with a minimum of 5 replicate analyses. Acceptance criteria are <5% RSD between the replicate analyses. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis, perform appropriate instrument maintenance then repeat.
- 10.5 *Internal Standards (ISTD)* are added to all standards, QC samples and environmental samples. Acceptance criteria vary by method as well as by sample type. Recovery is calculated by the instrument software and printed on the QC Calculated Values report for each sample.
- 10.5.1 Acceptance criteria for Method 200.8 are 60-120 %R, relative to the calibration standard, for all standards and samples.
- 10.5.2 Acceptance criteria for Method 6020A are >30 %R, relative to the calibration standard, for all samples analyzed.
- 10.5.3 If the acceptance criteria are not met, reanalyze the under a new calibration. If reanalysis fails reanalyze at a 1:2 dilution. If the diluted reanalysis fails prepare and analyze larger dilutions until the acceptance criteria are met.
- 10.6 A *Calibration Verification Standard* must be analyzed immediately after calibration, after every 10 samples, and after the last sample. The concentration of the ICV must be different than that of the CCV.
- 10.6.1 Recovery criteria are listed in the appropriate test code in LIMS. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate or report data with an appropriate qualifier.
- 10.6.2 ICV and CCV standards that fail to meet the acceptance criteria are automatically flagged in LIMS with an "S" qualifier.
- 10.6.3 The reporting of data associated with a failed control sample must be documented with a CAR form. If the failure is considered to have a significant affect on the data, client notification is required using the Case Narrative of the report.

- 10.6.4 Samples associated with a verification that fails with positive bias can be reported without narration if the sample concentration is below the reporting limit.
- 10.7 A *Calibration Verification Blank* sample must be analyzed after each calibration verification standard.
- 10.7.1 The acceptance criteria are < PQL. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate or report data with an appropriate qualifier. Samples for compliance with our Wisconsin DNR certification must be evaluated down to the current MDL and corrective action taken if the blank exceeds the routine PQL.
- 10.7.2 ICB and CCB standards that fail to meet the acceptance criteria are automatically flagged in LIMS with an "S" qualifier. ICB/CCB standards that are below the reporting limit but above the MDL are flagged in LIMS with a "b" qualifier. Data flagged with a "b" is considered as meeting the acceptance criteria.
- 10.7.3 If the blank does not meet the acceptance criteria, all samples < PQL or greater than 10-times the blank contamination may be reported. All other environmental samples must be reanalyzed or reported with an appropriate qualifier in the Case Narrative of the report. The reporting of data associated with a failed control sample must be documented with a CAR form.
- 10.7.4 An Instrument Check Standard (CAL STD 0.2 see 8.3.3.1) must be analyzed prior to the analysis of samples in each analytical run.
- 10.8 The *Interference Check Standards* (solutions A and AB) must be analyzed prior to the analysis of samples in each analytical run as well as every consecutive 12-hours of operation for Method 6020.
- 10.8.1 Acceptance criteria are the nominal limits listed in the appropriate test code in LIMS. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and recalibrate or report data with an appropriate qualifier.
- 10.8.2 The reporting of data associated with a failed control sample must be documented with a CAR form. If the failure is considered to have a significant affect on the data, client notification is required using the Case Narrative of the report.
- 10.8.3 Samples associated with a verification that fails with positive bias can be reported without narration if the sample concentration is below the reporting limit.
- 10.9 A *Method Blank* must be prepared and analyzed with each batch of maximum 20 samples and at a minimum of one per day.
- 10.9.1 The acceptance criteria are < PQL. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and reanalyze using a different calibration or report data with an appropriate

qualifier. Samples for compliance with our Wisconsin DNR certification must be evaluated down to the current MDL and corrective action taken if the blank exceeds the routine PQL.

- 10.9.2 MBLKs that fail to meet the acceptance criteria cause the sample results to be automatically flagged in LIMS with a "B" qualifier. MBLKs that are below the reporting limit but above the MDL are flagged in LIMS with a "b" qualifier. "b" flagged data is considered as meeting the acceptance criteria.
- 10.9.3 The reporting of data associated with a failed control sample must be documented with a CAR form. If the failure is considered to have a significant affect on the data, client notification is required using the Case Narrative of the report.
- 10.9.4 Samples associated with a MBLK that fails with positive bias can be reported without narration if the sample concentration is < PQL or greater than 10 times the blank contamination.
- 10.10 A *Laboratory Control Sample* must be prepared and analyzed with each batch of maximum 20 samples and at a minimum of one per day.
 - 10.10.1 Acceptance criteria are listed in the appropriate test code in LIMS. If the acceptance criteria are not met, reanalyze. If reanalysis fails to meet the acceptance criteria, stop analysis and reanalyze using a different calibration or report data with an appropriate qualifier.
 - 10.10.2 LCSs that fail to meet the acceptance criteria are automatically flagged in LIMS with an "S" qualifier.
 - 10.10.3 The reporting of data associated with a failed LCS must be documented with a CAR form. If the failure is considered to have a significant affect on the data, client notification is required using the Case Narrative of the report.
 - 10.10.4 Samples associated with a LCS that fails with positive bias can be reported without narration if the sample concentration is below the reporting limit.
- 10.11 A *Matrix Spike and Matrix Spike Duplicate* sample must be prepared and analyzed with each batch of maximum 20 samples per matrix and at a minimum of one per day.
 - 10.11.1 Acceptance criteria are listed in the appropriate test code in LIMS. (Note: the accuracy criteria have been met provided at least either the MS or MSD meet the %R criteria.) If the acceptance criteria are not met, refer to the MS/MSD Corrective Action Flowchart in the QAP.
 - 10.11.2 MS/MSD's that fail to meet the accuracy criteria are automatically flagged in LIMS with an "S" qualifier. MSD's that fail to meet the precision criteria are automatically flagged in LIMS with an "R" qualifier.

- 10.11.3 The reporting of data associated with a failed MS/MSD must be documented with a CAR form. If the failure is considered to have a significant affect on the data, client notification is required using the Case Narrative of the report.
- 10.11.4 Samples associated with a MS/MSD that fails the accuracy criteria with positive bias can be reported without narration if the sample concentration is below the reporting limit.
- 10.11.5 If the concentration measured in the sample is greater than 4-times the concentration of the spike, the spike amount used is insufficient and the MS/MSD not applicable.
- 10.12 A *Post Digestion Spike* can be analyzed on any sample to evaluate the potential of matrix interference.
- 10.12.1 Acceptance criteria are listed in the appropriate test code in LIMS.
- 10.12.2 The LIMS will flag all failed PDS recoveries with an "S" qualifier.
- 10.12.3 Use the recovery data to help evaluate any bias as detailed in the MS/MSD Corrective Action Flowchart.
- 10.13 A *Serial Dilution* can be analyzed on any sample to evaluate the potential of matrix interference (as indicated by failing IS/D recovery or failing MS/MSD/PDS recovery).
- 10.13.1 Acceptance criteria for a 1:5 dilution are $\leq 10\%$ difference between the original and the diluted sample concentration.
- 10.13.2 The LIMS will flag all failed SD recoveries with a "R" qualifier.

11.0 CALIBRATION AND STANDARDIZATION

Calibration data is documented and retained using the printouts from the instrument software. Analytical data must be maintained in accordance with the document control requirements in the Quality Assurance Plan as well as the Document Control SOP.

- 11.1 Repipetters must be calibrated/verified in accordance with the Repipetter Calibration SOP.
- 11.2 A new calibration must be performed for each analytical batch. Prepare the standards for the calibration curve as detailed in the Standards section of this SOP.
- 11.3 Perform the required preventative maintenance as necessary (indicated on Preventive Maintenance log). All preventative maintenance is documented in the Maintenance Log for the particular instrument used for analysis.

- 11.4 Turn on the exhaust hoods, vacuum pumps, recirculator, and empty the waste bucket. Set up the pump tubing for the sample, internal standard, and drain lines. Initiate the plasma and allow a minimum of 30-minutes for warm-up. During the warm-up time, the mass calibration and resolution checks may be performed.

11.5 TUNING and MASS CALIBRATION

- 11.5.1 Place the sample and internal standard lines into the Tuning Solution.

- 11.5.2 In the instrument software, open EPA200.8 Tuning workspace and click on the Tune toolbar. Clicking the Tune Mass Spec button will allow the instrument to automatically tune and mass calibrate.

- 11.5.3 Review the data.

- 11.5.3.1 For those masses having a resolution above 0.65amu, appropriately change DAC values to adjust the spectrometer resolution to produce a peak width of ≤ 0.65 amu at 10% of the peak height. (This is equivalent to 0.75amu @ 5% peak height meeting the requirements of both methods 200.8 and 6020.)

- 11.5.3.2 Appropriately adjust the mass calibration values for those masses not within 0.1amu of the unit mass. If adjustments were made, re-tune the instrument to verify appropriate and adequate operating conditions have been attained.

11.6 DAILY PERFORMANCE CHECK

- 11.6.1 With the sample and internal standard lines in the Tuning Solution, open the Daily Performance workspace and click on Analyze Sample in the sample window. When complete, the instrument will generate a Daily Performance Report.

- 11.6.2 Review the data and verify that the Net Intens. RSD results are $<5\%$ for all analytes (except "Bkgd").

- 11.6.3 Review the daily performance measurements or results against the instrument manufacturer recommended criteria below (Sensitivity, Background, %Double-charged, and %Oxide checks). If any adjustments are made due to failing the guidance criteria, repeat the daily performance test to verify appropriate and adequate operating conditions have been attained.

Sensitivity Checks

- Mg $>20,000$ cps for 10ppb
- Rh $>150,000$ cps for 10ppb
- Pb $>50,000$ cps for 10ppb
- If the sensitivity checks fail the cones may need cleaning or an AutoLens calibration may need to be performed.

Background Checks

- <30 cps at mass 220 for used skimmer cones. Brand new cones can give elevated counts at mass 220.
 - If the background check fails the detector voltage settings are too high.
- %Double-charged Checks**
- $Ba^{+2}/Ba \leq 3\%$
 - Failing double-charge checks are correctable by decreasing the nebulizer flow.
- %Oxides Checks**
- $CeO/Ce \leq 3\%$
 - Failing oxide checks are correctable by decreasing the nebulizer flow.

11.7 INTERNAL STANDARD MIXING CHECK

11.7.1 With the internal standard line in the Internal Standard solution and the sample line in a bottle of DI water, open the Internal Standard Mixing workspace and click on Analyze Sample in the sample window. When complete, the instrument will generate an Internal Standard Test Report.

11.7.2 Review the data and verify that the Int. RSD results are <3% for all internal standards [per Perkin-Elmer]. If the RSD criteria are not met, adjust the pump tube flow and repeat. Repeated adjustments are indicative of the need to replace the pump tubing.

11.8 DUAL DETECTOR CALIBRATION

11.8.1 Dual Detector Calibration is required only after changing detector voltages or installing a new detector.

11.8.2 With the internal standard line and the sample line in the Dual Detector Cross-Calibration Solution (see section 6.13 of the Perkin-Elmer method for 200.8 Using the ELAN 6000/6100/9000 ICP-MS), open the Dual Detector workspace and click on Optimization on the toolbar; go to the Dual Detector Calibration tab and clear the old calibration; choose Get Analytes then click Optimize. When complete, save the optimization file.

11.9 DAILY INSTRUMENT CALIBRATION

11.9.1 Open the 200.8 or 6020 workspace as appropriate.

11.9.2 Fill the autosampler tray with the appropriate standards and samples. The tube positions for the standards and samples are specified on the autosampler page of the analytical method. NOTE: all digestates must be diluted 1:5 for analysis on the mass spec (dilution provides the appropriate acid matrix).

11.9.3 Edit the sample window for batch analysis to update with the sample information.

- In the Batch ID column, enter the test code for the applicable sample

- In the Sample ID column, enter the LIMS sample ID or QC sample name in the order they are to be analyzed. Sample dilutions are also indicated in this field by adding a comma then the dilution factor (e.g. ME0607027-01A,5 indicates a 1:5 dilution of sample 7027-01A).
 - In the Measurement Action column, choose to run Blank, Standards, and Samples for the first sample to be analyzed but choose only Run Samples for the other samples to be analyzed
 - In the Method column, enter the method file to be used (e.g. EPA 200.8 Microbac or EPA 6020 Microbac)
- 11.9.4 Save the sample file with an appropriate name to indicate the date of analysis (e.g. "ICPMS-071008" for the analysis on 7/10/2008).
- 11.9.5 Select the samples to be analyzed by highlighting the row number with the mouse then select Analyze Batch. To avoid carryover and other potential contamination, aspirate the rinse blank approximately 5-10 minutes prior to beginning the batch analysis.
- 11.9.6 Following the instrument calibration, evaluate the calibration data using the instrument generated Calibration Report. Using a 1st Order Polynomial regression curve, the correlation coefficient must be $r \geq 0.995$. Based on the recommendation of the instrument manufacturer, the linear regression curve uses the "Linear thru zero" calibration option. Analysis of environmental samples cannot proceed without the generation of an acceptable calibration.
- 11.9.7 If the linearity requirements are not met, take appropriate corrective actions and recalibrate. If the acceptance criteria are met, continue with the analysis and evaluation of an ICV standard. The acceptance criteria must be met before continuing with sample analysis. Analysis of environmental samples cannot proceed without the generation of an acceptable linearity and an acceptable initial verification (see section 10 for details).
- 11.9.7 Unless you are running an unattended autosampler batch of samples, verify that the initial quality control (ICV, ICB, ICS, etc) is acceptable before continuing with sample analysis.

12.0 PROCEDURE

Analytical data is documented and retained using the printouts from the instrument software. Analytical data must be maintained in accordance with the document control requirements in the Quality Assurance Plan as well as the Document Control SOP.

12.1 Continue with the analysis of samples as initiated in the Calibration section.

12.2 Samples having a concentration above that of the high calibration standard (method 6020) must be diluted for reanalysis. Dilutions may be prepared directly in autosampler tubes using appropriate pipettes.

12.3 When the run is complete, the data is saved in C:\Elandata\Report Output\results.txt. Rename the results file with an appropriate name to indicate the

data of analysis (e.g. "ICPMS-071006" for the analysis on 7/10/2006) and copy it to the Instruments data server at I:\Metals\ICPMS.

- 12.4 Use the Combine and Save Report formatter to create a combined .pdf file of the various report files from the run. The individual files are saved in C:\Temp\0. The formatter combines the files and saves the new file to the server at P:\Instruments\Metals\ICPMS. Name the results file with an appropriate name to indicate the data of analysis (e.g. "ICPMS-071006.pdf" for the analysis on 7/10/2006)
- 12.5 Enter the data into LIMS. Refer to the SOP Analytical Data Entry – Metals for details.

13.0 CALCULATIONS AND DATA HANDLING

- 13.1 The instrument software calculates the concentration of the aqueous sample or digestate using linear regression ($y = mx + b$). This data is imported into LIMS during the Data Entry steps.
- 13.2 LIMS calculates the final sample concentration as follows:

$$\text{Concentration, mg/L} = (C) (0.001) (DF) (Pfac)$$

Where: C = concentration from curve, ug/L

DF = dilution factor of instrument

Pfac = (Final Volume, ml) / (Sample Size, ml or g)

- 13.3 The LIMS calculates the dry weight concentration for solid samples as follows:

$$\text{Conc. Dry} = \frac{(\text{wet weight conc.})}{(100 - \% \text{Moisture})}$$

- 13.4 Results flagged by the LIMS with an "E" qualifier are above the acceptable linear range. There is less certainty in these data and, if sufficient sample and holding time are available, should be reanalyzed at an appropriate dilution. Details on the procedure for entering analytical data are in the Analytical Data Entry - Metals SOP.

14.0 METHOD PERFORMANCE

- 14.1 Initial Demonstration of Capability study data, Method Detection Limit study data and Performance Testing study data are maintained and available from the QA office.

15.0 POLLUTION PREVENTION

- 15.1 The quantity of chemicals purchased should be based on expected usage during their shelf life and the disposal cost of unused material.

15.2 Prepare the minimum amount of reagent and standard necessary.

16.0 WASTE MANAGEMENT

16.1 Refer to the Sample Disposal SOP for guidance on the disposal of any resulting residue, digestate, distillate, extract or standard.

17.0 REFERENCES

17.1 USEPA Method 200.8, revision 5.4

17.2 SW-846 Method 6020A

17.3 SOP Preparation of Aqueous Samples and Extracts for Total or Dissolved Metals Analysis By Inductively Coupled Plasma, current revision.

17.4 SOP Preparation of Non-Aqueous Samples Total Metals Analysis, current revision.

17.5 SOP Toxicity Characteristic Leaching Procedure for Metals and Semi-Volatile Organic Compounds Using SW-846 Method 1311, current revision.

17.6 User's Manual EPA Method 200.8 Turnkey Method for the ELAN 6000, Perkin-Elmer Instruments, 2002 (see attached Table of Contents)

17.7 User's Manual EPA Method 6020 Turnkey Method for the ELAN 6000, Perkin-Elmer Instruments, 2002 (see attached Table of Contents)

17.8 Microbac Laboratories Quality Assurance Plan, current revision, all sections

18.0 TABLES, FORMS, CHECKLISTS, AND OTHER ATTACHMENTS

Copy of the Table of Contents from the Perkin Elmer 200.8 Turnkey method (1 page)

Copy of the Table of Contents from the Perkin Elmer 6020 Turnkey method (1 page)

Critical instrument method specifications (6 pages)

SOP Revision Notification form documenting the changes in this revision (1 page)

**EPA Method 200.8
Turnkey Method
For the ELAN[®] 6000/6100 ICP-MS**

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**EPA Method 6020
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Quantitative Method Report

File Name: epa 6020 microbac 08 LL.mth
File Path: C:\elandata\Method\epa 6020 microbac 08 LL.mth

Timing Parameters

Sweeps/Reading: 10
Readings/Replicate: 1
Number of Replicates: 3
Tuning File: c:\elandata\tuning\200.8 tuning.tun
Optimization File: c:\elandata\optimize\epa6020.dac
QC Enabled: Yes
Settling Time: Normal

Analyte	Mass	Scan Mode	MCA Channels	Dwell Time	Integration Time
Be	9.012	Peak Hopping	1	200.0 ms	2000 ms
B	11.009	Peak Hopping	1	200.0 ms	2000 ms
Al	26.982	Peak Hopping	1	100.0 ms	1000 ms
> Sc	44.956	Peak Hopping	1	100.0 ms	1000 ms
Ti	46.952	Peak Hopping	1	100.0 ms	1000 ms
V	50.944	Peak Hopping	1	100.0 ms	1000 ms
Cr	51.941	Peak Hopping	1	200.0 ms	2000 ms
Mn	54.938	Peak Hopping	1	100.0 ms	1000 ms
Co	58.933	Peak Hopping	1	100.0 ms	1000 ms
Ni	58.933	Peak Hopping	1	200.0 ms	2000 ms
Cu	62.930	Peak Hopping	1	100.0 ms	1000 ms
Cu	64.928	Peak Hopping	1	200.0 ms	2000 ms
Zn	65.926	Peak Hopping	1	200.0 ms	2000 ms
Zn	66.927	Peak Hopping	1	200.0 ms	2000 ms
Zn	67.925	Peak Hopping	1	200.0 ms	2000 ms
> Ge	71.922	Peak Hopping	1	100.0 ms	1000 ms
As	74.922	Peak Hopping	1	500.0 ms	5000 ms
Se	81.917	Peak Hopping	1	200.0 ms	2000 ms
Sr	87.906	Peak Hopping	1	100.0 ms	1000 ms
Mo	94.906	Peak Hopping	1	200.0 ms	2000 ms
Mo	97.906	Peak Hopping	1	200.0 ms	2000 ms
Ag	106.905	Peak Hopping	1	100.0 ms	1000 ms
Cd	110.90	Peak Hopping	1	200.0 ms	2000 ms
Cd	113.901	Peak Hopping	1	100.0 ms	1000 ms
> In	114.904	Peak Hopping	1	100.0 ms	1000 ms
Sn	117.902	Peak Hopping	1	100.0 ms	1000 ms
Sb	120.904	Peak Hopping	1	100.0 ms	1000 ms
Sb	122.904	Peak Hopping	1	100.0 ms	1000 ms
Ba	134.906	Peak Hopping	1	200.0 ms	2000 ms
Ba	136.905	Peak Hopping	1	100.0 ms	1000 ms
> Tb	158.925	Peak Hopping	1	100.0 ms	1000 ms

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Hg	201.971	Peak Hopping	1	500.0 ms	5000 ms
Tl	204.975	Peak Hopping	1	100.0 ms	1000 ms
Pb	207.977	Peak Hopping	1	100.0 ms	1000 ms
Na	22.990	Peak Hopping	1	100.0 ms	1000 ms
Mg	23.985	Peak Hopping	1	100.0 ms	1000 ms
K	38.964	Peak Hopping	1	100.0 ms	1000 ms
Ca	42.959	Peak Hopping	1	100.0 ms	1000 ms
Fe	53.940	Peak Hopping	1	100.0 ms	1000 ms
Fe	56.935	Peak Hopping	1	200.0 ms	2000 ms
Sc-1	44.956	Peak Hopping	1	100.0 ms	1000 ms
Kr	82.914	Peak Hopping	1	100.0 ms	1000 ms

Signal Processing

Detector Mode: Dual
 Measurement Units: cps
 AutoLens: On
 Spectral Peak Processing: Maximum
 Signal Profile Processing: Maximum
 Blank Subtraction: Subtracted after internal standard
 Baseline Readings: 0
 Smoothing: Yes, Factor 5

Equations

Analyte	Mass	Corrections
Al	26.982	-0.01 * C 13
Sc	44.956	-0.01 * Si 29
V	50.944	-3.127 * (Cl 35 - (0.113 * Cr 52))
Cr	51.941	-0.03 * C 13
Mn	54.938	-0.00005 * Fe 54
Ni	58.933	-0.0025 * Ca 43
Ge	71.922	-0.0001 * Ca 43
As	74.922	-3.127 * (Ar 40 - (0.815 * Se 82))
Se	81.917	-1.008 * (Kr 84)
Mo	94.906	-0.1096 * (Ru 101)
Cd	110.904	-1.0 * (Zn 66 - (0.712 * Pd 106))
Cd	113.904	-0.025826 * Sn 118
In	114.904	-0.14032 * Sn 118
Sb	122.904	-0.127189 * Te 125
Pb	207.977	+1 * Pb 206 + 1 * Pb 207
Fe	53.940	-0.028226 * Cr 52
Sc-1	44.956	-0.01 * Si 29

Calibration Information

Analyte	Mass	Curve Type	Sample Units	Std Units	Std 1	Std 2	Std 3	Std 4
Be	9.012	Linear Thru Zero	ug/L	ug/L	0.2	1	20	100
B	11.009	Linear Thru Zero	ug/L	ug/L		1	20	100

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Al	26.982	Linear Thru Zero	ug/L	ug/L				1	20	100
Sc	44.956	Linear Thru Zero	ug/L	ug/L						
Ti	46.952	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
V	50.944	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Cr	51.941	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Mn	54.938	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Co	58.933	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Ni	59.933	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Cu	62.930	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Cu	64.928	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Zn	65.926	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Zn	66.927	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Zn	67.925	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Ge	71.922	Linear Thru Zero	ug/L	ug/L						
As	74.922	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Se	81.917	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Sr	87.906	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Mo	94.906	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Mo	97.906	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Ag	106.905	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Cd	110.904	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Cd	113.904	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
In	114.904	Linear Thru Zero	ug/L	ug/L						
Sn	117.902	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Sb	120.904	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Sb	122.904	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Ba	134.906	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Ba	136.905	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Tb	158.925	Linear Thru Zero	ug/L	ug/L						
Hg	201.971	Linear Thru Zero	ug/L	ug/L	0.04	0.2		2	5	
Tl	204.975	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Pb	207.977	Linear Thru Zero	ug/L	ug/L	0.2		1	20	100	
Na	22.990	Linear Thru Zero	ug/L	ug/L				20	100	
Mg	23.985	Linear Thru Zero	ug/L	ug/L				20	100	
K	38.964	Linear Thru Zero	ug/L	ug/L				20	100	
Ca	42.959	Linear Thru Zero	ug/L	ug/L				20	100	
Fe	53.940	Linear Thru Zero	ug/L	ug/L				20	100	
Fe	56.935	Linear Thru Zero	ug/L	ug/L				20	100	
Sc-1	44.956	Linear Thru Zero	ug/L	ug/L						
Kr	82.914	Linear Thru Zero	ug/L	ug/L						

Analyte	Mass	Std 5	Std 6	Std 7	Std 8	Std 9	Std 10	Std 11	Std 12
Be	9.012	500							
B	11.009								
Al	26.982	500							
Sc	44.956								
Ti	46.952	500							

V	50.944	500
Cr	51.941	500
Mn	54.938	500
Co	58.933	500
Ni	59.933	500
Cu	62.930	500
Cu	64.928	500
Zn	65.926	500
Zn	66.927	500
Zn	67.925	500
Ge	71.922	
As	74.922	500
Se	81.917	500
Sr	87.906	500
Mo	94.906	500
Mo	97.906	500
Ag	106.905	
Cd	110.904	500
Cd	113.904	500
In	114.904	
Sn	117.902	500
Sb	120.904	500
Sb	122.904	500
Ba	134.906	500
Ba	136.905	500
Tb	158.925	
Hg	201.971	
Tl	204.975	500
Pb	207.977	500
Na	22.990	500 1e+004
Mg	23.985	500 1e+004
K	38.964	500 1e+004
Ca	42.959	500 1e+004
Fe	53.940	500 1e+004
Fe	56.935	500 1e+004
Sc-1	44.956	
Kr	82.914	

	AS Pos	Sample Flush	Sample Flush	Read Delay	Read Delay	Wash	Wash
0	1	25 s	-48 rpm	30 s	-24 rpm	45 s	-48 rpm
.2	2	25 s	-48 rpm	30 s	-24 rpm	60 s	-48 rpm
1	3	25 s	-48 rpm	30 s	-24 rpm	60 s	-48 rpm
20	3	25 s	-48 rpm	30 s	-24 rpm	60 s	-48 rpm
100	4	25 s	-48 rpm	30 s	-24 rpm	60 s	-48 rpm
500	5	25 s	-48 rpm	30 s	-24 rpm	60 s	-48 rpm
10000	6	25 s	-48 rpm	30 s	-24 rpm	180 s	-48 rpm
Standard 7		25 s	-48 rpm	10 s	-24 rpm	45 s	-48 rpm

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Standard 8	25 s	-48 rpm	10 s	-24 rpm	75 s	-48 rpm
Standard 9	25 s	-48 rpm	10 s	-24 rpm	75 s	-48 rpm
Standard 10	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 11	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 12	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 13	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 14	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 15	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 16	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 17	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 18	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 19	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 20	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 21	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 22	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 23	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 24	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 25	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 26	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 27	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 28	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 29	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm
Standard 30	25 s	-48 rpm	10 s	-24 rpm	55 s	-48 rpm

Reporting Options

Report Template for Printing: 6020 qc report.rop
 Send to Printer: Yes
 Report Template for File: C:\elandata\Report Options\6020 qc report.rop
 Send to File: Yes
 Report Filename: c:\elandata\Report Output\results.txt
 Create NetCDF File: No
 Send to Serial Port: No
 Port: COM1

Sampling Devices

Peristaltic Pump Control: Yes
 Autosampler: Cetac ASX-500
 Autosampler Tray File: c:\elandata\autosampler\ceasx500\as500b.try
 Sampling Device Type: (None)
 Dil. Factor: 10
 Dil. to Vol. (mL): 10
 1st Dil. Pos.: 1
 Probe Purge Pos.: 10

FIAS Program

Step	Read	Time	Pump 1	Pump 2	Valve	A/S Loc.	Sw 2	Sw 3	Sw 4
------	------	------	--------	--------	-------	----------	------	------	------

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Repeat Statement

HGA Program

Description:

Sample Volume: uL

Injection Temperature: C

Injection Speed:

Read delay: s

Closure delay: s

Modifier #1:

Modifier #2:

Step	Cell Temp	Ramp	Hold	Int. Flow	Gas Norm.	Gas Alt.	To Vent	To ICP	Read
------	-----------	------	------	-----------	-----------	----------	---------	--------	------

Pipet Seq.	Mod#1	Mod#2	Sample	Start Step	Wash	Rep. From	End S	Wash	Rep. To	# Rep
------------	-------	-------	--------	------------	------	-----------	-------	------	---------	-------



SOP Revision Notification / Annual Review Form

SOP Name METALS BY ICP-MS

☒ New Revision Old Revision # 0 New Revision # 1

- Summary of changes:
- Removed the method names from the title.
 - Sec. 8.2: (1) Updated the vendor info for hydrochloric acid and nitric acid; (2) Removed Triton X-100 from reagents list.
 - Sec. 8.3: (1) Added 0.05-ml of Boron to the Multi-element Intermediate Verification Standard; (2) Updated the sources for many standards; (3) Added many standards that were used in the procedure but not listed in this section; (4) Added prep information for the boron and silver standards and the gold, palladium, and platinum standards; (5) Sec. 8.3.3.1: Added CAL STD 0.2 and updated some preparation volumes and concentrations.
 - Sec. 9.2: To reflect current lab practices, the hold temperature for solids was changed to 0.1 to 6.0°C and the location was changed to the walk-in cooler.
 - Sec. 10.0: Removed the Linear Dynamic Range QC test.
 - Sec. 11.5.3.1: Clarified the width of the peak height when masses having a resolution above 0.65amu
 - Sec. 11.6.3: (1) Updated the metals and concentrations for background and sensitivity checks to reflect current lab practices; (2) Explained that there are different cps values depending on the newness of the skimmer cones.
 - Sec. 11.9.6: The linear regression curve uses the "Linear thru zero" calibration option.
 - Sec. 18: (1) Updated the critical instrument method specifications to reflect that the regression line is forced through the zero point; (2) Updated the correction factor for the analytical masses as per the method.

By signing below, I certify that I have been notified of the approval of a new revision to the above mentioned SOP. I realize it is my responsibility to read, understand and perform the procedure as set forth in this new revision.

Initials & Date	Initials & Date	Initials & Date
_____	_____	_____
_____	_____	_____
_____	_____	_____

Annual Review Current Revision # _____

By signing below, I certify that I have re-read, understand and agree to perform the current revision of the above mentioned SOP.

Initials & Date	Initials & Date	Initials & Date
_____	_____	_____
_____	_____	_____
_____	_____	_____

Form revised 01/18/06



Appendix C – Microbac Laboratories' NELAP Certification



STATE OF ILLINOIS
ENVIRONMENTAL PROTECTION AGENCY
NELAP - RECOGNIZED



ENVIRONMENTAL LABORATORY ACCREDITATION

is hereby granted to

MICROBAC LABORATORIES, INC.
250 WEST 84TH DRIVE
MERRILLVILLE, IN 46410

NELAP ACCREDITED
ACCREDITATION NUMBER #100435



According to the Illinois Administrative Code, Title 35, Subtitle A, Chapter II, Part 186, ACCREDITATION OF LABORATORIES FOR DRINKING WATER, WASTEWATER AND HAZARDOUS WASTES ANALYSIS, the State of Illinois formally recognizes that this laboratory is technically competent to perform the environmental analyses listed on the scope of accreditation detailed below.

The laboratory agrees to perform all analyses listed on this scope of accreditation according to the Part 186 requirements and acknowledges that continued accreditation is dependent on successful ongoing compliance with the applicable requirements of Part 186. Please contact the Illinois EPA Environmental Laboratory Accreditation Program (IL ELAP) to verify the laboratory's scope of accreditation and accreditation status. Accreditation by the State of Illinois is not an endorsement or a guarantee of validity of the data generated by the laboratory.

Ron Turpin
Manager
Environmental Laboratory Accreditation Program

Scott D. Siders
Accreditation Officer
Environmental Laboratory Accreditation Program

Certificate No.: 002414
Expiration Date: 01/30/2010
Issued On: 11/19/2009

State of Illinois
Environmental Protection Agency

Certificate No.: 002414

Towards the Certificate of Approval

Microbac Laboratories, Inc.
250 West 84th Drive
Merrillville, IN 46410

According to the Illinois Administrative Code, Title 35, Subtitle A, Chapter II, Part 186, ACCREDITATION OF LABORATORIES FOR DRINKING WATER, WASTEWATER AND HAZARDOUS WASTES ANALYSIS, the State of Illinois formally recognizes that this laboratory is technically competent to perform the environmental analyses listed on the scope of accreditation detailed below.

The laboratory agrees to perform all analyses listed on this scope of accreditation according to the Part 186 requirements and acknowledges that continued accreditation is dependent on successful ongoing compliance with the applicable requirements of Part 186. Please contact the Illinois EPA Environmental Laboratory Accreditation Program (IL ELAP) to verify the laboratory's scope of accreditation and accreditation status. Accreditation by the State of Illinois is not an endorsement or a guarantee of validity of the data generated by the laboratory.

Drinking Water, Inorganic

SM4500F-C, 18Ed

Fluoride

SM4500H-B, 18Ed

Hydrogen Ion (pH)

USEPA200.7R4.4

Arsenic

Cadmium

Copper

Sodium

Barium

Calcium

Magnesium

Beryllium

Chromium

Nickel

USEPA200.8R5.4

Antimony

Beryllium

Copper

Mercury

Thallium

Arsenic

Cadmium

Lead

Nickel

Barium

Chromium

Manganese

Selenium

USEPA200.9R2.2

Antimony

Lead

Arsenic

Selenium

Cadmium

Thallium

USEPA245.1R3.0

Mercury

USEPA335.4R1.0

Cyanide

USEPA353.2R2.0

Nitrate

Nitrite

USEPA365.1R2.0

Orthophosphate

Hazardous and Solid Waste, Inorganic

1010

Ignitability

1311

TCLP (Organic and Inorganic)

1312

Synthetic Precipitation Leaching Procedure

6010B

Aluminum

Antimony

Arsenic

State of Illinois
Environmental Protection Agency

Certificate No.:

002414

wards the Certificate of Approval

Microbac Laboratories, Inc.
250 West 84th Drive
Merrillville, IN 46410

Hazardous and Solid Waste, Inorganic

6010B

Barium

Beryllium
Chromium
Iron
Manganese
Potassium
Sodium
Vanadium

Cadmium
Cobalt
Lead
Molybdenum
Selenium
Strontium
Zinc

Calcium
Copper
Magnesium
Nickel
Silver
Thallium

6020A

Aluminum
Barium
Calcium
Copper
Magnesium
Molybdenum
Selenium
Thallium

Antimony
Beryllium
Chromium
Iron
Manganese
Nickel
Silver
Vanadium

Arsenic
Cadmium
Cobalt
Lead
Mercury
Potassium
Sodium
Zinc

7041 (Non-Potable Matrix Only)

Antimony

7060A

Arsenic

7131A

Cadmium

7196A

Chromium VI

7421

Lead

7470A

Mercury

7471A

Mercury

7841

Thallium

9012A

Cyanide

9030B

Sulfides

9034

Sulfides

9041A

Hydrogen Ion (pH)

9045C

Hydrogen Ion (pH)

9066

State of Illinois
Environmental Protection Agency

Certificate No.:

002414

Awards the Certificate of Approval

Microbac Laboratories, Inc.
 250 West 84th Drive
 Merrillville, IN 46410

Hazardous and Solid Waste, Inorganic

9066

Phenolics

9095A

Paint Filter

Chapter 7/9014

Reactive Cyanide

Chapter 7/9034

Reactive Sulfide

Hazardous and Solid Waste, Organic

8015B

Diesel range organics (DRO)

Gasoline range organics (GRO)

8081A

4,4'-DDD

Aldrin

Chlordane - not otherwise specified

Endosulfan I

Endrin

gamma-BHC (Lindane)

Methoxychlor

4,4'-DDE

alpha-BHC

delta-BHC

Endosulfan II

Endrin aldehyde

Heptachlor

Toxaphene

4,4'-DDT

beta-BHC

Dieldrin

Endosulfan sulfate

Endrin ketone

Heptachlor epoxide

8082

PCB-1016

PCB-1242

PCB-1260

PCB-1221

PCB-1248

PCB-1232

PCB-1254

8151A

2,4,5-TP (Silvex)

2,4-D

8260B

1,1,1,2-Tetrachloroethane

1,1,2-Trichloroethane

1,1-Dichloropropene

1,2,4-Trimethylbenzene

1,2-Dichlorobenzene

1,3,5-Trimethylbenzene

1,4-Dichlorobenzene

2-Hexanone

4-Methyl-2-pentanone (Methyl isobutyl ketone, I

Acrolein (Propenal)

Bromobenzene

Bromomethane

Chlorobenzene

Chloroform

cis-1,3-Dichloropropene

Ethylbenzene

m-Xylene

n-Butylbenzene

sec-Butylbenzene

Tetrachloroethene

1,1,1-Trichloroethane

1,1-Dichloroethane

1,2,3-Trichlorobenzene

1,2-Dibromo-3-chloropropane (DBCP)

1,2-Dichloroethane

1,3-Dichlorobenzene

2-Butanone (Methyl ethyl ketone, MEK)

2-Nitropropane

Acetone

Acrylonitrile

Bromodichloromethane

Carbon disulfide

Chlorodibromomethane (Dibromochloromethane)

Chloromethane

Dichloromethane (Methylene chloride)

Isopropylbenzene

Naphthalene

o-Xylene

Styrene

Toluene

1,1,2,2-Tetrachloroethane

1,1-Dichloroethene

1,2,4-Trichlorobenzene

1,2-Dibromoethane (EDB)

1,2-Dichloropropane

1,3-Dichloropropane

2-Chloroethyl vinyl ether

2-Pentanone

Acetonitrile

Benzene

Bromoform

Carbon tetrachloride

Chloroethane

cis-1,2-Dichloroethene

Ethyl acetate

Methyl-t-butyl ether

n-Butanol

p-Xylene

tert-Butylbenzene

trans-1,2-Dichloroethene

State of Illinois
Environmental Protection Agency

Certificate No.:

002414

towards the Certificate of Approval

Microbac Laboratories, Inc.
 250 West 84th Drive
 Merrillville, IN 46410

Hazardous and Solid Waste, Organic

8260B

trans-1,3-Dichloropropene

Trichloroethene

Trichlorofluoromethane

Vinyl acetate

Vinyl chloride

Xylenes (Total)

8270C

1,2,4-Trichlorobenzene

1,2-Dichlorobenzene

1,2-Diphenylhydrazine

1,3-Dichlorobenzene

1,4-Dichlorobenzene

2,4,5-Trichlorophenol

2,4,6-Trichlorophenol

2,4-Dichlorophenol

2,4-Dimethylphenol

2,4-Dinitrophenol

2,4-Dinitrotoluene (2,4-DNT)

2,6-Dichlorophenol

2,6-Dinitrotoluene (2,6-DNT)

2-Chloronaphthalene

2-Chlorophenol

2-Methylnaphthalene

2-Methylphenol

2-Nitroaniline

2-Nitrophenol

3,3'-Dichlorobenzidine

3-Nitroaniline

4,6-Dinitro-2-methylphenol

4-Bromophenyl phenyl ether

4-Chloro-3-methylphenol

4-Chloroaniline

4-Chlorophenyl phenyl ether

4-Nitroaniline

4-Nitrophenol

Acenaphthene

Acenaphthylene

Acetophenone

Aniline

Anthracene

Benzidine

Benzo(a)anthracene

Benzo(a)pyrene

Benzo(b)fluoranthene

Benzo(g,h,i)perylene

Benzo(k)fluoranthene

Benzoic acid

Benzyl alcohol

Bis(2-chloroethoxy) methane

Bis(2-chloroethyl) ether

Bis(2-ethylhexyl) phthalate

Butyl benzyl phthalate

Chrysene

Dibenz(a,h)anthracene

Dibenzofuran

Diethyl phthalate

Dimethyl phthalate

Di-n-butyl phthalate

Di-n-octyl phthalate

Fluoranthene

Fluorene

Hexachlorobenzene

Hexachlorobutadiene

Hexachlorocyclopentadiene

Hexachloroethane

Indeno(1,2,3-cd) pyrene

Isophorone

m-Cresol (3-Methylphenol)

Naphthalene

Nitrobenzene

N-Nitrosodimethylamine

N-Nitrosodi-n-propylamine

N-Nitrosodiphenylamine

p-Cresol (4-Methylphenol)

Pentachlorophenol

Phenanthrene

Phenol

Pyrene

Pyridine

Wastewater, Inorganic

SM2120B, 18Ed

Color

SM2320B, 18Ed

Alkalinity

SM2340B, 18Ed

Hardness

SM2340C, 18Ed

Hardness

SM2510B, 18Ed

Specific Conductance

SM2540B, 18Ed

Residue (Total)

SM2540C, 18Ed

Residue (TDS)

SM2540D, 18Ed

Residue (TSS)

State of Illinois
Environmental Protection Agency
Towards the Certificate of Approval

Certificate No.: 002414

Microbac Laboratories, Inc.
250 West 84th Drive
Merrillville, IN 46410

Wastewater, Inorganic

SM2550B, 18Ed

Temperature

SM3500Cr-D, 18Ed

Chromium VI

SM4500Cl-B, 18Ed

Chloride

SM4500CN-CE, 18Ed

Cyanide

SM4500CN-CG, 18Ed

Cyanide-amenable to chlorination

SM4500H-B, 18Ed

Hydrogen Ion (pH)

SM4500O-C, 18Ed

Oxygen - Dissolved

SM5210B, 18Ed

Biochemical Oxygen Demand (BOD)

Carbonaceous Biochemical Oxygen Demand (C

USEPA160.4

Residue (Volatile)

USEPA1631E

Mercury

USEPA1664RA

Oil and Grease

USEPA200.7R4.4

Aluminum

Antimony

Arsenic

Barium

Beryllium

Boron

Cadmium

Calcium

Chromium

Cobalt

Copper

Iron

Lead

Magnesium

Manganese

Molybdenum

Nickel

Potassium

Selenium

Silica

Silver

Sodium

Thallium

Tin

Vanadium

Zinc

USEPA200.8R5.4

Aluminum

Antimony

Arsenic

Barium

Beryllium

Boron

Cadmium

Chromium

Cobalt

Copper

Iron

Lead

Manganese

Molybdenum

Nickel

Selenium

Silver

Thallium

Vanadium

Zinc

USEPA200.9R2.2

Antimony

Arsenic

Cadmium

Lead

Selenium

Thallium

USEPA245.1R3.0

State of Illinois
Environmental Protection Agency
wards the Certificate of Approval

Certificate No.:

002414

Microbac Laboratories, Inc.
250 West 84th Drive
Merrillville, IN 46410

Wastewater, Inorganic	USEPA245.1R3.0	Mercury
USEPA350.1R2.0		
Ammonia		
USEPA353.2R2.0		
Nitrate-Nitrite (sum)	Nitrite	
USEPA365.1R2.0		
Orthophosphate (as P)		
USEPA410.4R2.0		
Chemical Oxygen Demand (COD)		
USEPA420.4R1.0		
Phenolics		
Wastewater, Organic		
USEPA608		
4,4'-DDD	4,4'-DDE	4,4'-DDT
Aldrin	alpha-BHC	beta-BHC
Chlordane	delta-BHC	Dieldrin
Endosulfan I	Endosulfan II	Endosulfan sulfate
Endrin	Endrin aldehyde	gamma-BHC (Lindane)
Heptachlor	Heptachlor epoxide	Methoxychlor
PCB-1016	PCB-1221	PCB-1232
PCB-1242	PCB-1248	PCB-1254
PCB-1260	Toxaphene	
USEPA610		
Acenaphthene	Acenaphthylene	Anthracene
Benzo(a)anthracene	Benzo(a)pyrene	Benzo(b)fluoranthene
Benzo(g,h,i)perylene	Benzo(k)fluoranthene	Chrysene
Dibenz(a,h)anthracene	Fluoranthene	Fluorene
Indeno(1,2,3-cd) pyrene	Naphthalene	Phenanthrene
Pyrene		
USEPA624		
1,1,1-Trichloroethane	1,1,2,2-Tetrachloroethane	1,1,2-Trichloroethane
1,1-Dichloroethane	1,1-Dichloroethene	1,2-Dichlorobenzene
1,2-Dichloroethane	1,2-Dichloropropane	1,3-Dichlorobenzene
1,4-Dichlorobenzene	2-Chloroethylvinyl ether	Acetonitrile
Acrolein (Propenal)	Acrylonitrile	Benzene
Bromodichloromethane	Bromoform	Bromomethane
Carbon tetrachloride	Chlorobenzene	Chloroethane
Chloroform	Chloromethane	cis-1,3-Dichloropropene
Dibromochloromethane	Dichloromethane (Methylene chloride)	Ethylbenzene
Tetrachloroethene	Toluene	trans-1,2-Dichloroethene
trans-1,3-Dichloropropene	Trichloroethene	Trichlorofluoromethane
Vinyl chloride	Xylenes (total)	
USEPA625		
1,2,4-Trichlorobenzene	1,2-Dichlorobenzene	1,3-Dichlorobenzene
1,4-Dichlorobenzene	2,4,6-Trichlorophenol	2,4-Dichlorophenol

State of Illinois
Environmental Protection Agency

Certificate No.:

002414

towards the Certificate of Approval

Microbac Laboratories, Inc.
250 West 84th Drive
Merrillville, IN 46410

Wastewater, Organic

2,4-Dinitrotoluene (2,4-DNT)
2-Chlorophenol
4-Bromophenyl phenyl ether
4-Nitrophenol
Anthracene
Benzo(a)pyrene
Benzo(k)fluoranthene
Bis(2-chloroethyl) ether
Dibenz(a,h)anthracene
Di-n-butyl phthalate
Fluorene
Hexachlorocyclopentadiene
Isophorone
N-Nitrosodimethylamine
Pentachlorophenol
Pyrene

USEPA625

2,6-Dinitrotoluene (2,6-DNT)
2-Nitrophenol
4-Chloro-3-methylphenol
Acenaphthene
Benzidine
Benzo(b)fluoranthene
Benzyl butyl phthalate
Bis(2-ethylhexyl) phthalate
Diethyl phthalate
Di-n-octyl phthalate
Hexachlorobenzene
Hexachloroethane
Naphthalene
N-Nitrosodi-n-propylamine
Phenanthrene

2,4-Dimethylphenol
2-Chloronaphthalene
3,3'-Dichlorobenzidine
4-Chlorophenyl phenyl ether
Acenaphthylene
Benzo(a)anthracene
Benzo(g,h,i)perylene
Bis(2-chloroethoxy) methane
Chrysene
Dimethyl phthalate
Fluoranthene
Hexachlorobutadiene
Indeno(1,2,3-cd) pyrene
Nitrobenzene
N-Nitrosodiphenylamine
Phenol

**XRF Sampling and Analysis Plan
for the
Quemetco Inc., Facility
Indianapolis, Indiana**

U.S. EPA ID# IND000199653

Prepared by
WSP Environment & Energy
2025 Gateway Place, Suite 435
San Jose, CA 95110

Prepared for
U.S. Environmental Protection Agency, Region V

December 11, 2009

Terry Cirone, Vice President of RSR, Facility Project Manager

Date

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Project Manager

Date

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Date

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Date

Hak Cho, U.S. EPA Region 5 RCRA Manager

Date

Jose Cisneros, U.S. EPA Region 5 RCRA Manager

Date

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Appendices

Appendix A – WSP Environment and Energy's SOP

Appendix B – Microbac Laboratories' SOP

Appendix C – Microbac Laboratories' NELAP Certification

1 Introduction

On behalf of Quemetco Inc. (Quemetco), WSP Environment & Energy (WSP) has prepared this XRF Sampling and Analysis Plan for the Quemetco facility located at 900 Quemetco Drive, in Indianapolis, Indiana (Site) (Figure 1). WSP prepared and submitted a revised Work Plan dated May 19, 2008 for additional sampling at the Quemetco facility. The Work Plan included collecting soil samples from the northeast area, slag waste pile area, around the ponds and along Juli a Creek. Surface water samples were to be collected from Pond #3 and Julia Creek. Collection of sediment samples were proposed for all three ponds and Julia Creek. The United States Environmental Protection Agency Region 5 (U.S. EPA) provided comments for the revised Work Plan in a letter dated October 8, 2009. In their letter, U.S. EPA requested that soil samples be collected and analyzed using X-ray fluorescence (XRF) technology. This Sampling and Analysis Plan was prepared in accordance with the U.S. EPA request. The letter describes a grid-space sampling approach for completing site-wide characterization and requests Quemetco perform investigative activities using X-ray fluorescence (XRF) for field sampling of lead. This Sampling and Analysis Plan describes the methodology for collecting and analyzing soil samples to further delineate the extent of soil impacted by lead.

The work described in this XRF Sampling and Analysis Plan will be performed in advance of revising the May 19, 2008 revised Work Plan and in accordance with U.S. EPA's letter dated October 8, 2009.

2 Soil Investigation

2.1 SAMPLING STRATEGY AND APPROACH

The investigation will consist of Surficial Zone and Subsurface Zone soil sampling. WSP estimates that up to 426 soil samples from approximately 284 locations will be collected during this investigation. Samples in each grid will alternate between (1) collection of a surficial sample (Surficial Zone) and (2) collection of both a surficial sample and a 0 to 6 inch depth sample (Subsurface Zone). Soil samples will be collected at 70 foot intervals from exposed soil in Zone-1, Zone-2, Zone-3, and Zone-4 of the Site (Figure 2).

Each of the four Zones contains a grid of squares 70 feet by 70 feet in size with the exception of some areas bordering the slag waste pile, ponds, and Julia Creek. Zone-1 includes the area north of the facility extending beyond the northern property boundary and the area west of the facility extending to the railroad berm. Zone-2 includes the area around Pond #3, bordering the slag waste pile to the west and Quemetco Drive to the east. Zone-3 starts at sample location NE-SS-31 and extends east to the western bank of Julia Creek and south to the southern fence line. Zone-4 includes the area around Pond #1 and Pond #2 and extends east to the western bank of Julia Creek.

Each sample location will be recorded using a GPS device. A map depicting all sample locations and surrounding areas will be provided with the final report. WSP will notify U.S. EPA at least 48 hours before any scheduled fieldwork. The following sections describe the sampling strategy, investigation methods and procedures, sample analyses, sample handling, decontamination procedures, and quality assurance and quality control.

2.1.1 Surficial Zone Soil Sampling

Surficial Zone soil sampling will be conducted at up to 284 locations. Surface soil samples for lead analysis will be collected in accordance with SW-846, Guidance Method 6200, section 11.3 (Feb. 2007). A dedicated hand trowel or stainless steel spoon will be used to collect surface soil samples. The trowels or spoons will be decontaminated before use. To minimize cross contamination, a new trowel or spoon will be used for each subsequent sampling location. Decontamination procedures are described in Section 2.5. Surficial zone sampling will be conducted as follows:

- Clear the sample area of debris, rocks, pebbles, and organic matter.
- The soil surface must be smooth to provide good contact with the soil sampling protection plate of the XRF device. If necessary, the surface will be leveled with a stainless-steel trowel or spoon.
- The surface will be tamped to increase soil density, based on the SW-846 protocol; this process will allow better repeatability and ensure that the sample is representative.
- The XRF does not perform well for saturated soils; WSP field representatives will ensure soil is not saturated prior to XRF analysis.
- The field lead analysis will be performed in accordance with the manufacturer's manual for operation of XRF instrumentation. The XRF device (along with a soil sampling protection plate) will be placed directly on the sampling location for a minimum of 60 nominal seconds (i.e., 60 seconds counted by the XRF device). Based on manufacturer's specifications, this method of analysis detects lead in the upper few millimeters of soil in contact with the soil protection plate.

The following sampling information will be recorded in a field notebook: sample zone location; sample GPS coordinates; sample identification numbers; date and time; sample depth; field lead concentration; and description of any visible evidence of soil contamination (i.e., odor, staining).

2.1.2 Subsurface Zone Soil Sampling

Subsurface soil samples for lead analysis will be collected in accordance with SW-846, Guidance Method 6200, Section 11.4 (Feb. 2007). Under Section 11.4, ex-situ soil samples should be homogenized, dried, and ground before XRF analysis. WSP contacted EPA requesting a variance for the homogenization and drying techniques described in Sections 11.4 and 11.5, respectively. With EPA's approval, WSP will not be utilizing fluorescent dye and ultraviolet light for homogenization or the drying technique using an oven and two to four hours of drying time.

Subsurface Zone soil sampling will be conducted at up to 142 locations. All subsurface soil samples will be collected at the same locations as the Surficial Zone sampling locations at a sample interval depth of 0 to 6 inches below ground surface (bgs). Subsurface soil samples for lead analysis will be collected with a dedicated hand trowel or stainless steel spoon. The trowels or spoons will be decontaminated before use. To minimize cross contamination, a new trowel or spoon will be used for each subsequent sampling location. Decontamination procedures are described in Section 2.5. Subsurface Zone sampling will be conducted as follows:

- Place the blade tip of trowel into the soil and push firmly until a sampling depth of approximately six inches is reached.
- Lift a portion of the soil out with the blade and place in a new plastic bag.
- Homogenize the sample in a plastic bag before drying.
- The field lead analysis will be performed in accordance with the manufacturer's manual for operation of XRF instrumentation. The XRF device will be placed directly on the plastic bags for a minimum of 60 nominal seconds according to manufacturer's specifications.
- For samples requiring fixed-laboratory analysis, transfer the plastic bag contents to a laboratory provided sample container using the sampling trowel or spoon.
- Label and place the sample in a cooler containing ice as described in Section 2.3.2.

After XRF analysis, 5% of sampled grids will be sampled for fixed-laboratory analysis of total lead according to the SW-846 guidance method using inductively coupled plasma-mass spectrometry (ICP-MS). As described above, all samples collected for fixed-laboratory analysis of total lead will be subsampled from the homogenized field sample. The field samples will be removed from plastic bags and placed into pre-cleaned, laboratory-supplied glass jars and stored in a thermally insulated cooler for transport to Microbac Laboratories.

The following sampling information will be recorded in a field notebook: sample zone location; sample GPS coordinates; sample identification numbers; date and time; sample depth; field lead concentration; and description of any visible evidence of soil contamination (i.e., odor, staining). Upon completion of all sampling activities, all sampling-holes will be backfilled with soil cuttings and compacted with a heavy blunt object.

2.2 SAMPLE ANALYSES

Soil samples collected at the Site will be analyzed by Microbac Laboratories (Microbac) at 250 West 84th Drive in Merrillville, Indiana using inductively coupled plasma-mass spectrometry (ICP-MS) listed in SW-846, Guidance Method 6200, Section 1.2 (Feb. 2007). Microbac is accredited by the National

Environmental Laboratory Accreditation Program (NELAP) (Appendix C). The following samples will be collected and analyzed as part of this investigation effort:

- Up to 15 Surficial Zone soil samples will be analyzed for lead by EPA method 200.8
- Up to 7 Subsurface Zone soil samples will be analyzed for lead by EPA method 200.8

Microbac will provide a CLP-level IV data package.

2.2.1 Quality Control Samples

Equipment Blanks

Dedicated sampling equipment will be used to collect samples at each location, for that reason, equipment blanks will not be collected for this investigation.

Trip Blanks

The use of trip blanks is typically limited to volatile organic compound (VOC) analysis. Because VOC analysis was not proposed for this investigation, trip blanks will not be analyzed for this investigation.

2.3 SAMPLE HANDLING PROCEDURES

2.3.1 Soil Sample Containers and Preservatives

The laboratory will provide soil sample containers. The containers will be pre-cleaned and will not be rinsed before sample collection.

2.3.2 Sample Packaging and Shipment

To identify and manage samples obtained in the field, a sample label will be affixed to each sample container. The sample labels will include the following information:

- Site name
- Zone number
- Sample identification number
- Sampler's initials
- Date and time of collection
- Preservative, if any

Following collection and labeling, samples will be immediately placed in a sample cooler for temporary storage. The following protocol will be followed for sample packaging:

1. Samples to be shipped will be placed in the cooler and packed with packaging materials to minimize the potential for disturbance and/or breakage of the sample containers.
2. Ice or "Blue Ice" packs will be placed in leak-resistant plastic bags and added to the cooler s to chill the samples during transportation to the analytical laboratory.
3. The chain-of-custody form will be placed in a water-resistant plastic bag and taped on the inside of the lid of the cooler.

All samples will be maintained in the custody of the sampling team. The samples will be transported to the analytical laboratory at the end of the sampling day under appropriate chain-of-custody procedures.

2.4 SAMPLE DOCUMENTATION

2.4.1 Field Logbooks

Field logbooks will document where, when, how, and from whom any vital project information was obtained. Logbook entries will be bound with consecutively numbered pages. Each page will be dated and the time of entry noted in military time.

At a minimum, the following information will be recorded during the collection of each sample:

- Zone number
- Sample identification number
- GPS location coordinates
- Date and time of sample collection
- Type of sample (i.e., matrix)
- Type of sampling equipment used
- Field observations and details important to analysis or integrity of samples (e.g., heavy rains, odors, colors, etc)

2.4.2 Chain-of-Custody Records

Chain-of-custody (COC) records are used to document sample collection and shipment to the laboratory for analysis. All sample shipments will be accompanied by a COC record. The COC record will identify the contents of each shipment and maintain the custodial integrity of the samples.

2.5 DECONTAMINATION PROCEDURES

All equipment that comes into contact with potentially contaminated soil will be decontaminated before use. Disposable or dedicated equipment intended for one time use will not be decontaminated, but will be packaged in Quemetco's PPE refuse containers for appropriate disposal. All non-disposable sampling devices will be decontaminated using the following procedures:

- Non-phosphate detergent and tap water wash, using a brush if necessary
- Tap-water rinse

2.6 INVESTIGATION WASTE MANAGEMENT

In the process of collecting environmental samples during the proposed field sampling program, different types of potentially contaminated investigation-derived wastes (IDW) will be generated. The wastes will be handled as follows:

- Soil will be placed back into their respective sample-holes.
- Decontamination fluids will be processed through the wastewater treatment plant.
- Used personal protective equipment (PPE) and disposable equipment (used sampling trays) will be placed in Quemetco, Inc.'s PPE refuse containers. Used PPE will be handled as hazardous waste consistent with Quemetco, Inc.'s waste handling practices.

3 Reporting

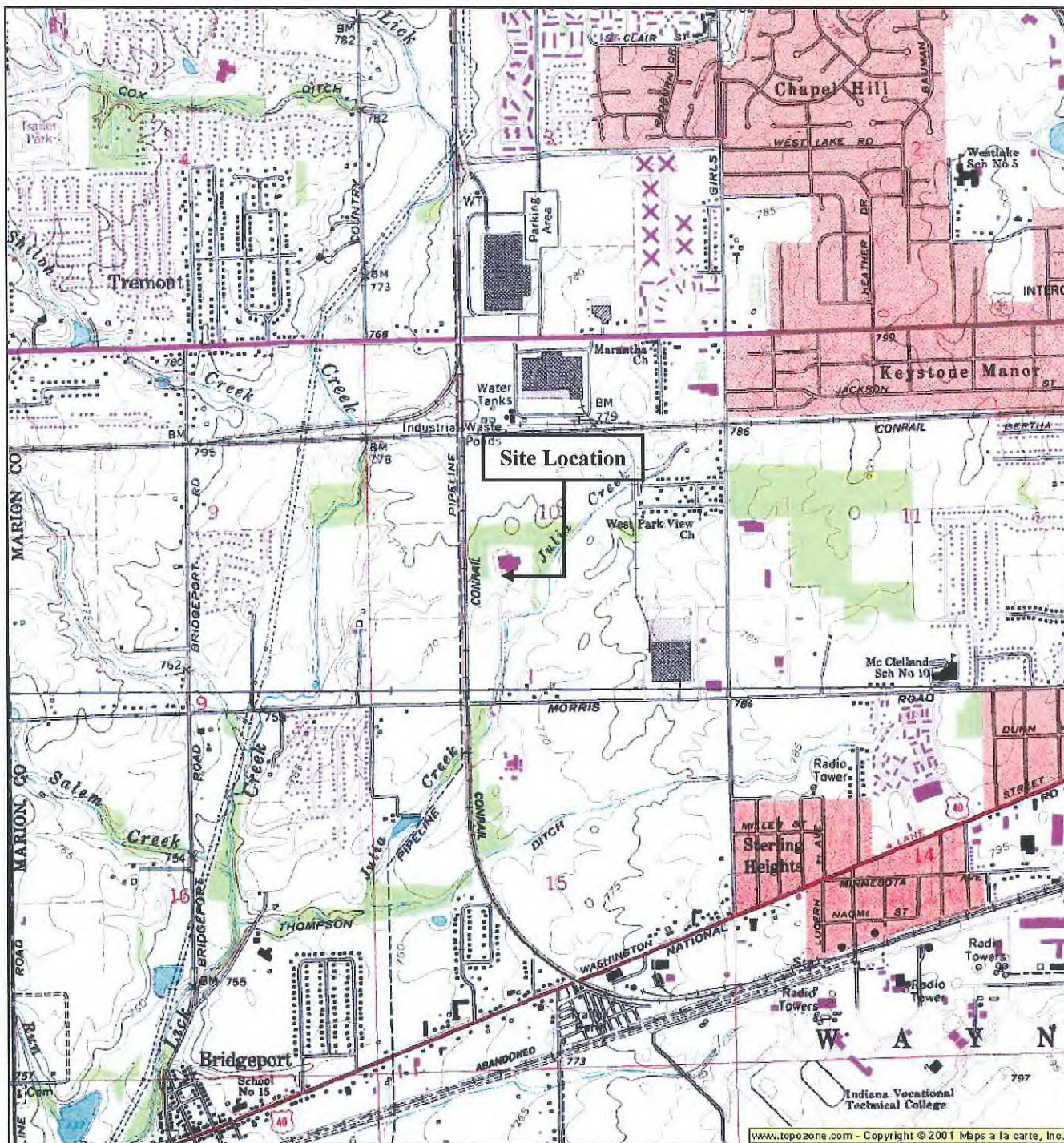
Upon completion of the sampling activities, a report detailing the investigation efforts will be prepared and submitted to U.S. EPA. The report will include sample methodology, summarized sample results, laboratory analytical and quality assurance and quality control review, and a map depicting GPS-recorded sample locations.

Terrie Baranek of ECT.CON Inc., will be the Quality Assurance Officer (QAO) during the project and will be responsible for all QA/QC aspects of the work, including data validation of both field and laboratory data. The QAO will remain independent of direct job involvement and day-to-day operations, report to the WSP Project Director, and resolve any QA disputes.

Following a review of the data collected according to this Sampling and Analysis Plan, WSP will prepare a revision to the May 19, 2008 revised Work Plan to address any remaining data gaps and U.S. EPA's comments in their letter dated October 8, 2009.



Figure 1 – Site Location



Reference

7.5 Minute Series Topographic Quadrangle
Clermont, Indiana
Photorevised 1984 Scale 1:25,000



1000 0 2000 400
Scale in Feet

Quadrangle Location



WSP Environment & Energy
2025 Gateway Place, Suite 435
San Jose, California
(408) 453-6100

Figure 1
Site Location
Location of Quemetco Facility
Indianapolis, Indiana



Figure 2 – Sampling Grid for XRF